User Meeting 2014

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National Centre for Synchrotron Science

Book of Abstracts

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Advanced Materials I - Sponsored by AXAA / 6

In-situ Small Angle X-ray Scattering Investigation of Formation of Meso-porous Silica Nanoparticles and Swelling-Shrinking Growth Mechanism

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The general explanation of how MSNs grow is that the silica monomers hydrolysed from silica precursor adsorbed onto surfactant micelles and condensed into silica to form particles. However, the detailed growth mechanism of MSNs still remains unknown especially for the MSNs with small particle size. Herein, time-resolved SAXS with a synchrotron source was employed to investigate the growth of MSNs under different experimental conditions. The fitting models were introduced into the system to interpret the SAXS data. The results suggested that the added TEOS inserted into the hydrophobic core of CTAB micelles, forming an emulsion at the beginning of the growth. The silica monomers were hydrolysed from the inside of the micelles into the solvent. The CTAB micelles were actually swelled by the addition of TEOS and with the consumption of the TEOS the pores formed by CTAB was shrunk to form the final meso-pores. To the best of our knowledge, this mechanism is found for the first time, which will highly contribute to the understanding of MSN growth mechanism.

Keywords or phrases (comma separated):

mesoporous silica nanoparticles, growth mechanism, time-resolved SAXS

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Competition in phase formation during crystallisation of Al-Ni-Y metallic glasses

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Metallic glasses have attracted substantial attention in recent years due to their favourable combinations of high strength and corrosion resistance relative to conventional crystalline alloys. These properties make glassy metals appealing for applications such as surgical tools, electronics and sporting goods. However, glassy metals are metastable and crystallisation occurs when they are subjected to elevated temperatures or sustained deformation. While crystallisation is often considered detrimental to the properties, in some cases, controlled crystallisation can produce novel microstructures with unusual and desirable combinations of properties. The effect of crystallisation depends on which phases form, and the order of their appearance.

Of the Al-based metallic glasses, the ternary Al-Ni-Y system is among the most well studied. However, the sequence of phases that form during crystallisation remains unclear. In this investigation the crystallisation pathways in four Al-Ni-Y alloys with Ni concentrations ranging from 9 to 15 at.%

have been studied in detail by in situ synchrotron powder diffraction. These experiments reveal that at low Ni concentrations crystallisation occurs via a two stage process, with &alpha-Al forming as the first decomposition product, while at high concentrations crystallisation occurs via a three stage process, with the metastable Al9Ni2 phase forming first. The level of detail afforded by this approach allows us to better understand the competition in phase formation during crystallisation of metallic glasses and to use this information in the design of thermal treatments and compositions to optimize their potential usefulness.

Keywords or phrases (comma separated):

metallic glasses, crystallisation, in situ, powder diffraction

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Characterization of nanoscale precipitates in a new 2GPa strength steel using small angle x-ray scattering

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Steels are used extensively in the manufacture of automobiles. They may not sound like advanced materials to those not involved in their development – after all they have been available for centuries. However, if we consider a typical modern automobile, none of the steels found in the structure existed 10 years ago. These are engineering alloys that are being intensively developed and improved, and more so than any other alloy class.
This presentation discusses a new class of steel with strengths approaching 2GPa that was developed for applications in the automotive industry. The alloy design process included the coupling of computational thermodynamics with a Genetic Algorithm for compositional optimization. The dominant contribution to these high strengths is a nanoscale distribution of particles within the material and a quantitative understanding of their size, shape and volume fraction is a key requirement for rationalizing the observed properties.
The particle distribution was characterized at the Australian Synchrotron using small angle x-ray scattering (SAXS) in both ex-situ samples and during in-situ thermal treatments to monitor the earliest stages of particle nucleation and growth. Combined with 3D measurements of particle compositions using atom probe tomography (APT), it was possible to extract bulk quantitative measures of the particle volume fractions and hence calculate representative number densities of particles. These nanoscale particles have number densities comparable to the highest densities so far observed in engineering alloys. The information gained from these SAXS and APT experiments helps to verify and improve the computational alloy design process.

Keywords or phrases (comma separated):

SAXS, steel, nanoscale precipitation

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Materials Characterisation and X-ray Free Electron Laser Science at the Centre of Excellence for Advanced Molecular Imaging

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With the recent availability of X-ray Free Electron Lasers (XFELs) and the prospect of Diffraction Limited Storage Rings (DLSRs) just around the corner, the number of major new scientific breakthroughs in the area of coherent X-rays science is likely to rise sharply over the next few years. The past twelve months has already seen significant progress in the field including 3D imaging of intragrain deformation in polycrystals, the study of hysteretic behavior in solid solution and two-phase reactions within nanoparticles and the development of fly-scanning coherent diffractive imaging combined with fluorescence mapping.

Over the last 8 years our group has contributed to experimental and theoretical developments within the field of Coherent Diffractive Imaging (CDI) which are now finding a number of key applications. In particular our work in the areas of partial coherence and diffractive imaging using curved beams have emerged as being two particularly important contributions to the field. Here we present some of our recent work in developing CDI for the mapping of deformation within nanocrystals, characterising Medium Range Order (MRO) and in exploring the limits of partial coherence in diffractive imaging. As part of the newly funded ARC Centre of Excellence for Advanced Molecular Imaging we plan to apply these methods to key problems in biology both at the synchrotron and XFEL. We will also briefly discuss the implications for CDI of the new DLSR upgrades potentially taking place at the ESRF, Spring-8 and APS within the next 5-10 years.

Keywords or phrases (comma separated):

Coherent X-ray Science

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IR spectroscopy of ferrocene and deuterated ferrocene: Experiment and theory

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The contemporary organometallic chemistry stems from the discovery of ferrocene Fe(C5H5)2, i.e., di-cyclopentadienyle iron (FeCp2 or Fc) half a century ago. Since its discovery, the heated debate whether the eclipsed or the staggered is the most stable structure of Fc continues. The fact that electronic structures and many properties of the Fc conformers are strikingly similar has been a key hurdle to differentiate or separate the configurations from one another. We recently discovered theoretically using DFT calculations that the 400-500 cm-1 region of the infrared (IR) spectra of Fc exhibits the fingerprint conformers. Such the discovery was later confirmed by IR experimental measurements in a number of solutions, in polar and non-polar solvents. Understanding of the structure and dynamics of the sandwich complex is important as Fc derivatives may inherit particular properties which only exist in one conformer such as catalysis. It is further discovered that the IR spectral fingerprint is associated with the vibrations of the centre Fe atom in the sandwich conformers, which is seen in an earlier IR experiment of Fc. As a result, we designed and conducted new high-resolution IR experiments in the gas-phase Far-IR beamline of the Australian Synchrotron to study Fc-h10 and deuterated Fc-d10. New results and analysis in gas phase and in solutions will be presented at this meeting.

Keywords or phrases (comma separated):

IR spectroscopy, ferrocene and it isotopes, DFT calculations

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Synchrotron Powder Diffraction Study of Cement Pastes

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The degree of hydration of cement pastes is critical for determining properties such as the durability of concrete. As part of an integrated study on the prediction of chloride ingress in reinforced concrete, synchrotron X-ray powder diffraction was used to estimate the degree of hydration of cement pastes. While for the past 20 years the composition of Portland cement has been determined by Rietveld analysis of X-ray diffraction, nevertheless there are a number of factors, including the amorphous content of the cement and relative proportion of mineral polymorphs present in the initial clinker, whose impact on the analysis are still not completely understood. X-ray powder diffraction beamlines from The Brazilian Synchrotron Light Laboratory (LNLS) and The Australian Synchrotron were used to analyze a suite of production cements from both countries. The results showed significant differences in degree of hydration and composition in the two cements.

Keywords or phrases (comma separated):

Engineering, Cement, Concrete, Infrastructure, Durability

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New Packings and Properties for Supramolecular Nanoballs through Solvent and Counterion Variation

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We have synthesised large (2.7 nm) spherical metallosupramolecules (‘nanoballs’) with interesting properties [1-3]. Metal ions can be varied with retention of overall structure and crystal packing. The molecular packing creates cavities within the solid state, and the crystals readily absorb solvents such as methanol, acetonitrile or acetone (which also changes the magnetic properties), and absorb significant amounts of hydrogen and carbon dioxide (but not methane), pointing to a new class of porous materials. Other properties include switching between two magnetic spin states (spin crossover) upon change in temperature or irradiation of light, and size-selective catalysis. New packing arrangements of the nanoballs can then be achieved through variation of the counteranions or nitrile solvent, leading to new phases with different physical properties.

[1] M.B. Duriska, S.M. Neville, B. Moubaraki, et al., Angew. Chem. Int. Ed., 2009, 48, 2549.
[2] M.B. Duriska, S.M. Neville, J. Lu, et al., Angew. Chem. Int. Ed., 2009, 48, 8919.
[3] M.B. Duriska, S.M. Neville, B. Moubaraki, et al., ChemPlusChem, 2012, 77, 616.

Keywords or phrases (comma separated):

Supramolecular Chemistry, Nanoballs, Chemical Crystallography

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In Situ PXRD Studies of the Solvothermal Syntheses of WO₃-Ethylenediamine Hybrid Nanowires and Bi₂Se_xTe_{3-x} Nanoplatelets

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Solvothermal syntheses are very versatile for fabricating nanostructured materials. While the majority of studies focus on materials syntheses, little attention has been paid to understanding the synthesis mechanisms, which are of vital importance to the rational design of synthesis for preparing optimized materials. In this context, in situ powder X-ray diffraction (PXRD) is ideal to study solvothermal materials syntheses as it is capable of providing direct reaction information under the harsh autoclave synthesis conditions. In this presentation, I will show the application of in situ PXRD in solving the mechanisms of the solvothermal synthesis of (1) WO3-ethylenediamine inorganic-organic hybrid nanowires, and (2) Bi2SexTe3-x nanoplatelets. The WO3-ethylenediamine nanowires showed excellent performance in catalysis and water treatment and Bi2SexTe3-x nanoplatelets are topological insulators that have potential applications in the electronic and optoelectronics areas. In the in situ PXRD experiments, solvothermal syntheses were conducted in quartz glass microreactors that were placed at the beam centre of the Australian Synchrotron powder diffraction beamline, and the time resolved PXRD patterns were recorded to follow the phase evolution during the synthesis. For both materials, in situ PXRD discovered intermediate phases that played an important role in controlling the formation of the final nanostructured materials.

Keywords or phrases (comma separated):

In situ PXRD, solvothermal synthesis, nanowires, nanoplatelets

Beamline updates and discussion / **142**

XAS beamline update

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In this session we will update on the status of the XAS Beamline, including developments of the user science program, status of the 100-element fluorescence detector, status of Hutch-C, recent works completed, and further plans for upgrades and developments. The session will provide ample opportunity for Q A and for discussing science needs that the XAS Beamline is not catering for, such as the Medium Energy XAS (MEX) Beamline.

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The XFM beamline: Status, upgrades and directions, enabling user science across a range of disciplines

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The X-Ray Fluorescence Microscopy beamline has hosted around 250 user groups since it started user operations in 2008, and sustains high levels of oversubscription. Armed with the world-leading Maia detector, and ongoing collaboration with CSIRO, almost 100,000 scans have been performed, representing an estimated 100 Gpixels of data and a stage transit of around 200 km with a positioning accuracy of 2 m.
In order to harness the power of the Maia detector, we have undertaken an ambitious series of upgrades. This presentation describes the beamline instrumentation with reference to applications, and recent upgrades to the table, scanning stages, tomography, large-area scanning, cryogenics, and configurable specimen mounting stages, as well as beamline ancillaries including equipment for specimen pre-alignment and cryogenic specimen preparation. It will then close with a short exploration of the future directions for the beamline's evolution.

This session will include an extended open discussion for XFM users.

Keywords or phrases (comma separated):

X-ray fluorescence microscopy, XANES, tomography, upgrades, status

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Absolute Dosimetry using a Graphite Calorimeter on the Imaging and Medical Beamline at the Australian Synchrotron

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In 2012-13, the Australian Synchrotron installed a superconducting multi-pole wiggler (SCMPW) on the imaging and medical beamline (IMBL) with the intention of future preclinical radiotherapy trials. Before moving to such trials, accurate knowledge of the dose delivered is required to calibrate the secondary dosimeters in regular use. When this is resolved, clinical use of the Australian Synchrotron will be closer to reality.
The dose rate achievable on the IMBL is in the range 10 Gy/s to 10 kGy/s, depending on the SCMPW magnetic field, the filtration set and the distance of the measurement rooms (or hutches) from the photon source. High-dose-rate dosimetry is challenging with detectors such as free-air ionization chambers (FAC), due to the high ion-recombination corrections required. An absolute method of dose determination is required and one such method is graphite calorimetry, which is based on the temperature rise in graphite when irradiated. In this meth-od, the temperature is measured by a calibrated thermistor embedded in the calorimeter graphite core. The ab-sorbed dose from the irradiation is determined from the product of the temperature rise and the specific heat capacity of graphite, corrected for the ratio of graphite core area to beam area.
Results will be presented for measurements in hutches 1B, 2B and 3B, where the dose rates presently vary from 20 Gy/s to 3000 Gy/s. Comparisons with a free-air chamber and calculated dose rates will be presented.

Keywords or phrases (comma separated):

Graphite, Calorimetry, Absolute Dosimetry

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Characterisation of the PTW microDiamond detector for high spatial resolution dosimetry in microbeam radiation therapy at IMBL

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Microbeam radiation therapy (MRT) at IMBL is based on arrays of 50&mu m wide x-ray beams with a pitch of 400&mu m. The peak-to-valley dose ratio (PVDR) is the ratio of the peak dose to the dose between the microbeams and is an important radiobiological quantity. Accurate measurements of the PVDR require a dosimeter with high spatial resolution, dose rate independence and water equivalence for the MRT spectrum. The PTW microDiamond detector is a synthetic single crystal diamond detector. The 1.1mm radius and 1&mu m thickness make it a promising candidate MRT dosimetry. Studies have been performed at IMBL to characterise the energy, dose rate and directional dependence of microDiamond. The ratio of mass energy absorption coefficients in diamond and water predict that the detector will under-respond at low energies, however, this was not observed in the experimental ratio of the microDiamond response to absorbed dose in water for energies 30-90keV. The dose rate dependence was found to be linear for storage ring currents &ge 50mA but deviated from linearity by up to 4% for lower currents (2-50mA). The reponse of the detector oriented at 0o and 90o relative to normal beam incidence agreed to within 3%. This is an important result since the required spatial resolution for PVDR measurements exists in the 90o geometry. The detector will be calibrated against reference detectors and the kV primary standard for absorbed dose.

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X-ray Imaging at IMBL: Detailed Considerations of Contrast and Resolution

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The Imaging and Medical Beamline (IMBL) at the Australian Synchrotron has three hutches, centred at 22, 36 and 140m from the source, in which user experiments are performed. Radiotherapy experiments are currently performed in the first of these hutches, and imaging (including tomography) in the second and third hutches. The X-ray source is provided by a superconducting multipole wiggler (SCMPW) insertion device. A double-crystal Laue monochromator (DCLM) can be used to select the

desired X-ray energy within the range 20 - 120keV at present. Alternatively, a white (pink) beam can be employed, using appropriate in-vacuo filters.

We will provide a detailed description and analysis of the key factors which influence the quality of X-ray images which can be recorded. In addition to the nature of the sample itself, the factors considered include the SCMPW field, X-ray energy or spectrum used, source size, detector resolution, source-to-sample and sample-to-detector distances. The key parameters used in describing the "quality" of an X-ray image are contrast (both absorption and phase) and resolution. An objective assessment of the point-spread function will be central and some discussion of signal-to-noise ratio will also be included. Other factors such as the presence of a small harmonic-contamination contribution for certain operation of the DCLM will be considered.

Keywords or phrases (comma separated):

Imaging, Tomography, Contrast, Resolution

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Spatial dosimetric response of an ionisation chamber to kilovoltage synchrotron radiation by 2D scanning in a sub-millimetre beam

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The IMBL at the Australian Synchrotron is able to provide high-brightness kilovoltage radiation in the energy range 20 – 200 keV. A PTW 30013 Farmer-type ionisation chamber was scanned through a point-like (sub-millimetre) beam and the ionisation signal from the chamber was recorded as a function of position. In this way an image was constructed from the spatial dosimetric response of the chamber. Such information can be used to determine, for example, the fraction of response from the chamber stem to the overall response of the chamber. Other interesting features include the contribution from the aluminium central electrode, and increased response where the walls are side-on to the beam (and therefore contribute a greater number of secondary electrons to the air cavity). The results are compared to a Monte Carlo model. Dosimetric response maps should be useful for investigating the design of ionisation chambers for radiotherapy.

Keywords or phrases (comma separated):

ionisation chamber, radiotherapy, dosimetry

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Chemical Crystallography at the Australian Synchrotron MX Beamlines

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The Macromolecular Crystallography (MX) Beamlines at the Australian Synchrotron collect data on protein samples (PX) and chemical samples (CX). This broad range of sample types requires us to consider a number of experimental and data processing considerations. Protein samples have very large unit cells but diffract weakly, the chemical samples on the other hand have comparatively a very small unit cell, diffract much more strongly and to higher resolution. From an experimental point of view, this requires substantially different geometrical considerations which can be handled by changing the energy of the monochromated X-rays and detector distance. Another consideration is detector type, the detectors at the MX beamlines are from the Area Detector Systems Corporation (ADSC) and they have generally been used for PX data collections.
As the detectors have mainly been used for PX work, the software for sample handling has also been developed with PX considerations rather than CX. For example, the XDS software for space group determinations is set by default to assume that the space group is only ever one of the 65 space groups that don't contain mirror, inversion or glide operations. Another area of interest is the way data scaling (absorption) is handled. The data is often scaled with PX data for a number of reasons, with the most common scaling of data is due to the prevalence of radiation damage to the samples. By contrast the most common form of scaling for CX data is for sample anisotropy in strong absorbers.

Keywords or phrases (comma separated):

chemical, crystallography, synchrotron, detector

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Crystallography beamline update - what is going on at MX?

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The Macromolecular Crystallography (MX) beamlines serve the scientific community by enabling research that could not be done elsewhere in Australia. In order to achieve this, we are in a state of continual improvement. Here we present an overview of recently completed, currently being rolled-out, and near future developments at MX1 and MX2. Projects that have been recently completed include a major upgrade of the sample mounting robots; new filter wheels for fine control beam attenuation; And roof-mounted HD gimbal cameras inside the experimental hutches. Future plans include a more reliable and user-friendly interface for remote access; and improvements to the area for incoming shipping dewars. Looking forward, a major upgrade to the MX2 optics is at hand during the next shutdown, involving a new channel-cut Si-crystal for improved beam-stability and a piezo-collimator for producing micro-beams at higher flux and with greater control than the current micro-collimator.

Keywords or phrases (comma separated):

crystallography, macromolecular, chemical, small molecule, instrumentation

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Broader horizons for Bragg Coherent Diffractive Imaging; energy scanning and time-resolved measurements

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Bragg Coherent Diffractive Imaging (BCDI) is a technique which is rapidly gaining in popularity throughout the X-ray microscopy community. BCDI allows the characterisation of both the shape and three-dimensional deformation field of nanocrystals at spatial resolutions approaching a few nm. Typically BCDI is sensitive to displacements of < 10-4 of a lattice spacing (Abbey, JOM, 2013). Measurements are normally performed by 'rocking' a single crystal through the Bragg condition whilst recording the fine structure around one of the Bragg peaks. Current data acquisition times are on the order of tens of minutes, making the study of non-reversible processes in dynamically evolving systems extremely challenging. We have recently shown that scanning the monochromator energy such that the crystal moves in and out of the diffraction condition provides equivalent BCDI information to rocking the crystal. Building upon this idea we show here that illuminating the nanocrystal with the broadband radiation from a single undulator peak and scanning the undulator gap provides a significant increase in the available flux with a concomitant reduction in the data acquisition time. We plan to use this technique to study systems which are evolving on timescales of minutes, perhaps even seconds allowing BCDI to be applied to study a range of time-resolved processes including annealing.

Keywords or phrases (comma separated):

Bragg CDI, Partial Coherence, Strain Mapping

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The THz/Far-IR Beamline at the Australian Synchrotron

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The THz/Far-IR beamline at the Australian Synchrotron is coupled to a Bruker IFS125 FT spectrometer which is equipped with a variety of optical components and detectors covering the spectral range from 10 to 5000 cm-1. Experiments from a variety of fields such as atmospheric and astrophysical sciences, geology, electrochemistry, nano-materials as well as biology have been successfully conducted at the beamline.
There is a variety of instruments to accommodate the diverse requirements of the User community: long-path gas-cells to study gases, radicals generated by pyrolysis and aerosols; 6.3 K and 77 K cryostats to study condensed-phase samples in transmission, and reflection studies at grazing incidence, as well as near-normal incidence at high-temperatures (< 1000 K).
The synchrotron terahertz and far-infrared light offers a signal-to-noise advantage over conventional thermal sources. In this paper, the capabilities and performance of the THz/Far-IR beamline at the Australian Synchrotron will be presented as well as some applications undertaken at the beamline, and future developments.

Keywords or phrases (comma separated):

THz, Far-IR, spectroscopy, gas-phase, condensed-phase, synchrotron, beamline

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Probing materials at 100 nm resolution by AFM-based near field FTIR

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Developments to enable infrared microspectroscopy to extend beyond the far field diffraction limit are being undertaken at several accelerator facilities worldwide. These include the CLIO Free Electron Laser (Paris, France), LNLS (Campinas, Brazil), and the ALS infrared beamline (Berkeley, USA). Without such developments, the spatial resolution in the mid-IR is typically 3 to 5 microns. Two alternative techniques based on photothermal expansion (CLIO) and on near-field scattering from an AFM probe (LNLS, ALS) are used by these facilities, and a beamline dedicated to this technique is planned for LNLS, with potential to push the spatial resolution limit to less than 100 nm. The IR beamline group at the Australian Synchrotron have gained experience of both methods, through successful beamtime at CLIO and at the ALS. Results from the most recent beamtime at the ALS AFM-IR beamline instrument show the potential to collect representative IR spectra from sub micron samples which had not been achievable using the IR beamline at the Australian Synchrotron. Details of the instrumentation at the ALS beamline will be described, along with results from samples, including the detection surface glucan molecules on the hyphae of Candida albicans fungi. Such instrumentation could potentially be installed as an add-on to the IRM beamline at the Australian Synchrotron.

Keywords or phrases (comma separated):

FTIR, near field, AFM, infection

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Zinc complex rescues subcellular zinc and calcium mislocalisation in batten disease

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A hallmark of neurodegeneration is a failure of homeostatic mechanisms controlling the concentration and distribution of biometals. A major roadblock to understanding the impact of altered biometal homeostasis in neurodegenerative disease is the lack of specific and sensitive techniques capable of providing quantitative subcellular information on biometals in situ. Advances in X-ray fluorescence detectors provide an opportunity to rapidly measure biometal content at subcellular resolution in cells using X-ray Fluorescence Microscopy (XFM). We investigated subcellular biometal homeostasis in a cerebellar cell line from a natural mouse model of a childhood neurodegenerative disorder, the CLN6 form of Batten Disease. Despite no global cell concentration changes, XFM revealed significant subcellular mislocalisation of zinc and calcium in cerebellar Cln6nclf cells. XFM revealed that nuclear-to-cytoplasmic trafficking of zinc was severely perturbed in diseased cells and the subcellular distribution of calcium was drastically altered in Cln6nclf cells. Subtle differences in the zinc XANES spectra of control and Cln6nclf cells suggested that impaired zinc homeostasis may be associated with an altered ligand set in Cln6nclf cells. Importantly, a zinc-complex, ZnII(atsm), restored the nuclear-to-cytoplasmic zinc ratios in Cln6nclf cells via nuclear zinc delivery, and restored the relationship between subcellular zinc and calcium levels to that observed in healthy control cells. ZnII(atsm) treatment also resulted in a reduction in the number of calcium-rich puncta observed in Cln6nclf cells. This study highlights the complementarities of bulk and single cell analysis of metal content for understanding disease states.

Keywords or phrases (comma separated):

Zinc, XFM, neurodegeneration, calcium, XANES, biometal homeostasis, CLN6

Biological Systems / 4

Mechanisms of action of a potent DNA binding UVA photosensitiser using mRNA-sequencing and infrared Synchrotron microspectroscopy

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Co-author(s): Danielle Martin [2] ; Katherine Ververis [1] ; Li-Jeen Mah [1] ; Stephanie Tortorella [1] ; Virat Ganthavee [1]

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Phototherapy is a well-established therapeutic strategy in dermatology, particularly for the treatment of psoriasis and cutaneous T-cell lymphoma. Treatment may either rely on the cytotoxic effect of light of a particular wavelength (e.g. UVB and narrowband UVB) or may require the use of a sensitizer (e.g. psoralens with UVA). We have developed iodinated DNA minor groove binding bisbenzimidazoles as UVA sensitizers. We investigated the phototoxicity of a number of iodinated bibenzimidazoles and UVASens, proved to be outstanding with respect to photopotency, due in part to the very high quantum yield of photodeiodination. Indeed, the photopotency of the iodinated bibenzimidazole is about 1000-fold higher than that of psoralens. We have used genome-wide mRNA-Sequencing and infrared Synchrotron microspectroscopy to gain insights into the mechanisms accounting for the phototoxicity of UVASens in human erythroleukemic K562 cells. Infrared spectra indicate unique signatures for cells treated with combinations of UVASens and UVA light compared to untreated cells and cells treated with either UVASens or UVA light alone. Analysis indicates that mechanisms of cytotoxicity involve inhibition of DNA synthesis, lipid peroxidation and induction of apoptosis. Further, mRNA-sequencing reveals changed expression of >8,000 genes following treatment of cells with combinations of UVASens and UVA with many of those genes involved in pathways regulating cell cycle, cell-death and apoptosis. Overall, our findings highlight the extreme photopotency of UVASens. We are currently investigating the potential of specifically targeting malignant cells using UVASens loaded nanoparticle formulations.

Keywords or phrases (comma separated):

cutaneous T-cell lymphoma, UVA, phototherapy, DNA binding ligand

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Correlative single cell Fourier transform infrared spectroscopy and super-resolution fluorescence microscopy reveals the effects of fixation on the biochemistry of mammalian cells.

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Co-author(s): Toby Bell [1]

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Synchrotron Fourier transform infrared (SFTIR) spectroscopy provides the best available signal-to-noise which allows for fast, sensitive detection of the holistic biochemistry of single live cells. Recently developed super-resolution fluorescence microscopy (SRFM) techniques based on single molecule emissions can yield an order of magnitude improvement in spatial resolution in the imaging of specifically targeted cellular structures. For the first time we have paired these two techniques

to investigate the effects of various fixation parameters on the FTIR spectrum, the rendered super-resolution image, and the underlying biochemistry. To achieve this SFTIR spectra of single live COS7 cells were obtained; the cells were then fixed and a second spectrum of each hydrated correlated cell acquired. Finally, the cells were immunostained and the microtubule architecture visualized using direct stochastic optical reconstruction microscopy (dSTORM) with spatial resolutions of 20-30 nm achieved. The resulting FTIR spectra demonstrated that many of the spectral changes previously associated with cell fixation effects were due to dehydration, morphological variation and loss of cell constituents through washing. Key spectral changes that could be directly linked to the fixation parameters included changes in lipid content and ordering, DNA conformation, and glycogen and cytosolic protein concentration. Importantly, while FTIR spectroscopy was found to detect various subtle changes in the underlying biochemistry that SRFM could not, subdiffraction damage to the cell cytoskeleton could only be detected using SRFM. Not only does this work demonstrate the exquisite sensitivity of both techniques but also the future potential for correlative SFTIR/SRFM.

Keywords or phrases (comma separated):

FTIR, fluorescence, microscopy, fixation, super resolution, single cell,

Closing Session / **145**

Science Case II - status and future plans

Closing Session / **18**

Closing Remarks

Closing Session / **16**

X-ray Absorption Spectroscopy in an Age of Insertion Devices

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The first dedicated beamline for X-ray Absorption Spectroscopy (XAS) was built in 1974, since then XAS has become one of the core competencies of synchrotron radiation facilities and an essential tool for a broad range of scientific disciplines. XAS is a technique that remains closely associated with its roots in bend magnet sources and second generation facilities. In recent years, insertion device beamlines, often built on wiggler sources, have offered significant new measurement capabilities which capitalize on the increased flux offered by these insertion devices. Some beamlines, most notably ID26 at ESRF, use the high flux of the insertion device to replace conventional integrating or energy dispersive detectors with wavelength dispersive spectrometers. These crystal spectrometry systems both enhance the XAS capabilities of the beamline and make available other inner shell spectroscopic techniques such as X-ray Emission Spectroscopy and X-ray Raman Spectroscopy.

Even with such exciting developments in photon delivery and photon detection, conventional XAS beamlines remain highly productive, scientifically relevant, and popular to the point of substantial over-subscription. In this talk, I will introduce these novel spectroscopic techniques and discuss how they can complement a conventional XAS research campaign, in some cases providing specific details unavailable using conventional XAS or other measurement techniques. Finally, I will provide a brief introduction to the Inner Shell Spectroscopy beamline being built at the new completed National Synchrotron Light Source II.

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UAC Town Hall Meeting

Earth and Environment / 10

Distribution of aluminium in plant roots: Understanding its toxicity through correlative microscopy

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Aluminium (Al) is toxic to plant root growth in the acid soils comprising ca. 40-70% of the world's arable land, but the mechanisms whereby Al reduces growth remain unclear. Despite 30 μM Al decreasing root growth within 30 min, we are unaware of any study that has provided information on the distribution of Al in roots within this timeframe. Using roots of soybean (Glycine max (L.) Merr.) exposed to 30 μM Al, we identified and separated individual mechanisms of toxicity through the use of high resolution kinematic analyses together with the complementary use of synchrotron-based low-energy X-ray fluorescence spectromicroscopy (LEXRFS) and nano secondary ion mass spectroscopy (nanoSIMS). The latter techniques revealed the majority of Al to be located in the outer cellular layers within 30 min of exposure, decreasing markedly with increasing distance from the root surface. At the sub-cellular level, the majority of the Al was found to accumulate in the cell wall. Of particular importance, Al was bound strongly by (i) pectin in the corner junctions between cells, with this typically the location where Al first accumulated in the inner tissues, (ii) walls of the border cells (c.f. the walls of the adjacent rhizodermal cells), and (iii) the mucigel on the root surface. For the first time, correlative microscopy (LEXAFS and nanoSIMS) and other complementary techniques have provided important evidence as to where Al accumulates in the short term and how Al rapidly reduces the growth of plant roots.

Keywords or phrases (comma separated):

Correlative microscopy, Metal toxicity, Plant roots

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Chemical Speciation Imaging using Fast X-ray Fluorescence Microscopy: Update on capabilities and future directions

Author(s): David Paterson[1]

Co-author(s): Barbara Etschmann [2] ; Chris Ryan [3] ; Daryl Howard [1] ; Enzo Lombi [4] ; Erica Donner [4] ; Kathryn Spiers [1] ; Martin de Jonge [1] ; Robin Kirkham [3]

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[4] *University of South Australia*

X-ray fluorescence microscopy (XFM) can be used for elemental and chemical microanalysis across many length scales and is a powerful tool for quantitatively mapping trace elements within whole biological specimens [1]. Advances in X-ray fluorescence detection schemes [2, 3] now enable acquisition at mega-pixel per hour rates which in turn allows collection of 3D information in realistic times. Chemical speciation imaging (CSI) results in an image stack with the third dimension containing a XANES spectra in each pixel [4]. Fitting of spectra with incident X-ray beam energy tracking has been developed in GeoPIXE software using the Dynamic Analysis method [5, 6]. CSI has been demonstrated with moderate definition (10,000s of pixels/image) across a diverse range of applications [7, 8]. Recent studies have improved the efficiency and sensitivity of CSI to environmentally relevant concentrations.

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[6] C. G. Ryan et al., J. of Physics: Conf. Series 499, 012002 (2014).
[7] P. M. Kopittke, et al., New Phytologist 201, 1251 (2014).
[8] B. E. Etschmann et al., Environmental Chemistry, 11, 341 (2014).

Keywords or phrases (comma separated):

Chemical Speciation Imaging, X-ray Fluorescence Microscopy

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From Clouds to CFCs: IR spectroscopy of atmospheric and interstellar molecules.

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Co-author(s): Andrew Wong [2] ; Chris Medcraft [2] ; Courtney Ennis [3] ; Dominque Appadoo [4] ; Don McNaughton [2] ; Mahmut Ruzi [1]

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The IR spectral region is crucial to our understanding of atmospheric chemistry and physics. Firstly, radiative processes associated with emission and extinction are fundamental to energy transfer and the IR region in particular is central for materials at the moderate temperatures found in non-stellar environments such as this planet's atmosphere. Secondly, infrared spectroscopy is uniquely suited to remotely probing the properties of atmospheric molecules and aerosols.

We have employed the various facilities on the far IR beamline to advance understanding of atmospheric molecules such as HFCs and CFCs, and also aerosols such as water ice nanoparticles found in high altitude clouds. Our studies of crystalline water ice particles have recently been extended to both amorphous ice and to deuterium enriched crystalline ice, providing insights into fundamental questions about the water vibrations in these phases. An overview will be presented.

Keywords or phrases (comma separated):

IR spectroscopy, earth's atmosphere, water ice, CFCs, HFCs.

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Carbon speciation in soil: Effects on carbon turnover and carbon sequestration

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[3] *The University of Queensland*

[4] *Department of Science, Information Technology, Innovation and the Arts, Queensland Government*

Carbon storage in soil is essential for soil productivity while being directly linked to climate change. Mapping microaggregate-associated forms of soil organic carbon can help understanding the mechanisms of carbon stabilization in soil, revealing molecular organization, physical protection in soil particles and co-localization of carbon sources with microbial processes.
Spatially-resolved analyses of carbon distribution in microaggregates (<200 μm diameter) was conducted using FTIR microspectroscopy (Infrared Microspectroscopy beamline, Australian Synchrotron). Two soil types (Ferrosol and Vertosol) were collected from undisturbed areas and from locations immediately adjacent which have a long history of agricultural use. Soils were gently screened (250 μm) to obtain intact microaggregates which were humidified and frozen at -20℃, and sectioned (200 μm thickness) using a diamond knife and a cryo-ultramicrotome. The sections were placed between CaF2 windows and the spectra were acquired in transmission mode.
The maps obtained (5 μm step-size over ca. 150 × 150 μm) revealed for the first time carbon distribution in microaggregates from soils under contrasting land management. Accumulation of aromatic and carboxylic functions on specific spots and marginal co-localization with clays was observed, which suggests processes other than organo-mineral associations being responsible for carbon stabilization. A substantial decrease in carboxylic compounds was observed for agricultural soils. Clays were mostly co-localized with alkenes and polysaccharides, particularly in agricultural soils, likely due to enhanced microbial activity in those spots.
Results will be linked to currently ongoing analysis of enzymatic activities and dissolved organic carbon for further interpretation.

Keywords or phrases (comma separated):

Infrared Microspectroscopy; soil microaggregates; soil organic carbon; carbon sequestration;

Energy Materials / 9

Quantitative analysis of spotty diffraction rings and application to corrosion studies of steel

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[1] *Callaghan Innovation*

Spotty diffraction rings are symptomatic of large-grained polycrystalline materials. Analysis of these rings falls into a void between single crystal and powder diffraction methods, and is usually dismissed or discussed only briefly and in a qualitative fashion. Recently, we have developed statistical methods for quantitatively analysing the ‘spottiness’ of diffraction rings, such as those observed during CO2 corrosion of steel in aqueous solutions under electrochemical control. These include the normalised roughness and the fractal dimension of the diffraction ring, obtained from 2D diffraction images. Statistical measures obtained for diffraction patterns calculated from theoretical crystallite size distributions are compared to those obtained for experimental data. This illustrates that the corrosion mechanism relies on the formation of surface roughness, which proceeds via preferential dissolution of small grains.

References:
B. Ingham, ‘Statistical measures of spottiness in diffraction rings’, J. Appl. Cryst. 2013, 47, 166-172.

M. Ko, B. Ingham, N. Laycock and D. E. Williams, 'In situ synchrotron X-ray diffraction study of the effect of microstructure and boundary layer conditions on CO2 corrosion of pipeline steels', Corrosion Sci. (submitted).

Keywords or phrases (comma separated):

XRD, diffraction, analysis, corrosion

Energy Materials / 69

Using Flexible MOFs to Study Inorganic Reactivity

Chris Sumby[1]

[1] *University of Adelaide*

Metal-organic frameworks (MOFs) are crystalline materials that can be synthesised from metal ions or metal-oxide clusters (nodes) and organic building blocks (links).[1] Through careful consideration of the chemistry of the organic links the properties of these materials can be tailored for particular applications. For example, a flexible framework capable of high yielding post-synthetic metallation[2] can be synthesised.[3] Remarkably, this material (MnMOF) is able to provide structural insight into inorganic reactivity through single crystal X-ray crystallography (SCXRD).[3]

This presentation will describe the structure of this new 3D MOF, which possesses pore cavities that are lined with vacant di-pyrazole groups poised for post-synthetic metallation. As part of this I will illustrate the potential of this platform MOF to provide fundamental insight into metal-catalysed reactions in porous solids. For example, SCXRD studies undertaken partly at the Australian Synchrotron, reveal the reaction products of consecutive oxidative addition and methyl migration steps that occur within the pores of a Rh metallated example, MnMOF-[Rh(CO)2][Rh(CO)2Cl2].

[1] H. Furukawa, K.E. Cordova, M. O'Keeffe and O.M. Yaghi, Science, 2013, 341, 974.
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[3] W.M. Bloch, A. Burgun, C.J. Coghlan, R. Lee, M.L. Coote, C.J. Doonan, and C.J. Sumby, Nat. Chem., 2014, doi:10.1038/nchem.2045.

Keywords or phrases (comma separated):

MOFs, structural inorganic chemistry, post-synthetic reactions, organometallic chemistry

Energy Materials / 120

Smart adsorbents for gas separation research at Australian Synchrotron

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Zeolite molecular sieves are one of the most important materials for separation of molecules. We discovered smart porous materials for gas separation – zeolites containing cations that function as molecular trapdoors allow guest-selective, size-inverse separations. For example, a "molecular trapdoor" mechanism in specifically tailored zeolites which produces a counter-intuitive size-inverse "sieving" for CO/N2, and a record high selectivity for CO2/CH4 separation over a large pressure

range. [1] In the other case, we found an unusual operating regime on a chabazite zeolite in which the adsorption selectivity for N2 over CH4 inverts from being more selective for N2 at 253 K, to becoming less selective with increasing temperature and eventually becoming selective for CH4 over N2 above 293 K. [2] These materials could benefit for carbon capture and gas purification.
[1] Shang, Jin; Li, Gang; Singh, Ranjeet; Gu, Qinfen; et al. JOURNAL OF THE AMERICAN CHEMICAL SOCIETY,134, 46,19246-19253, (2012)
[2] Shang, Jin; Li, Gang; Gu, Qinfen; et al. CHEMICAL COMMUNICATIONS, 50, 35, 4544-4546, (2014)

Keywords or phrases (comma separated):

powder diffraction, zeolite, gas separation, structure

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Mechanistic and structural investigation of Li_xMnO₂ cathodes during cycling in Li-ion batteries

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Increasingly there is demand for clean energy sources and suitable batteries to store this energy. Manganese dioxide and lithiated variants are a promising alternative to conventional Li-ion cathodes due to their cost, abundance, safety and electrochemical performance. Cathodes which operate by a single-phase lithium insertion/extraction process can offer some intrinsic advantages over those with two-phase processes. In this work, in-situ and ex-situ synchrotron X-ray diffraction (XRD) is used to investigate the structural evolution and lithium insertion/extraction mechanism of various LixMnO2 cathodes that have been derived from γ-MnO2. Li0.30MnO2 is found to cycle solely with a single-phase mechanism, in contrast to previous literature reports, with only subtle changes in the crystal structure. However, a better cycling discharge capacity is realised through a two-step lithiation synthesis, thermally lithiated Li0.08MnO2 which is then electrochemically lithiated to Li0.33MnO2. After an irreversible two-phase reaction early in the first discharge, this material cycles by a single-phase reaction with good structural reversibility and a stable unoptimised cycling capacity of 120 mAh/g. Comparing cathodes using a combination of in-situ and ex-situ synchrotron XRD data allows us to rationalise cathodic performance with structure and thereby directing research into promising candidates.

Keywords or phrases (comma separated):

Manganese dioxide, in-situ synchrotron XRD, Li-ion, energy storage

Imaging - Sponsored by MASSIVE / 79

Dual Energy X-ray Analysis Using Synchrotron Computed Tomography at 35–60 keV

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Dual energy X-ray analysis (DEXA) uses CT measurements of the X-ray linear attenuation coefficient at two photon energies to characterise materials; electron density and statistical measure of elemental composition, related to the concept of effective atomic number.

Phantoms were prepared as liquid samples of known density and composition including ethanol-water mixtures and salt solutions (NaCl, NaH2PO4, MgCl2, MgSO4, KCl, KH2PO4 and CaCl2). The phantoms and an ex-vivo laboratory animal underwent mono-energetic CT at 35–60 keV using the Australian Synchrotron Imaging and Medical beamline and a CCD camera optically coupled to a luminescent screen.

The CT data for the phantoms provided coefficients that describe attenuation as measured by the beamline, and expressed as elemental cross-sections. The phantom data underwent DEXA, and the accuracy of the analysis was quantified as a function of mean beam energy, dual energy separation, and elemental composition. The CT data for ex-vivo samples was spatially co-registered, subjected to DEXA, and the results were used to create volumetric maps representing the X-ray linear attenuation coefficients and energy absorption coefficients for photon energies of 10 keV to 10 MeV.

The DEXA technique was successfully demonstrated using samples of known density and composition, and factors that affect accuracy were identified. An important application of the method, the prediction of photon interaction coefficients at other beam energies for attenuation correction and radiation dose calculations, was investigated for the biological specimen.

Keywords or phrases (comma separated):

synchrotron CT, electron density, composition, attenuation correction, radiation dosimetry

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Progress on the CT program at the Imaging and Medical beamline

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The Imaging and Medical Beamline (IMBL) of the Australian Synchrotron (AS) is now becoming one of the most advanced instruments of this type in the world. It is designed to provide a wide variety of imaging techniques. Three beamline's enclosures at various distances provide the end user a good choice of beam characteristics ranging from the hi-flux for lower resolution and size up to huge 48x5cm beam at 134m from the source with the allowed energy range 17-120kEv. The wide range of the area detectors at the beamline allows the computed tomography (CT) to be combined with almost any known X-ray imaging modality. The beamline's data acquisition system is directly linked to the high performance computing facility: MASSIVE tuned for the on-the-fly real-time reconstruction and 3D volume rendering. Advanced CT experiment control system with the unprecedented level of flexibility allows users to implement many of their ideas and construct an absolutely new experiment logic with minimal efforts and time. Deep integration of the acquisition, reconstruction and rendering facilities allows one to think of the their combination as of a a single system with modular architecture. This report summarizes implemented, designed and planned features of the beamline as applied to the CT type of the experiment. Brief overview of the CT experiments conducted in this year is given. Some latest outcomes of the CT system are presented with the samples coming of different fields of science: biology, geology, paleontology, material science and medicine.

Keywords or phrases (comma separated):

computed tomography, X-ray imaging, biology, geology, paleontology, medicine.

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High Resolution Imaging and Strain Characterisation at Pulsed Neutron Sources with a Microchannel plate detector

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Recent advances in neutron detection technology are enabling collection of neutron transmission data with unprecedented spatial and time resolution [1,2]. Microchannel plates coupled with TimePix area detectors are now being used to perform time-of-flight neutron radiography experiments at 55 micron2 spatial resolution and 1 &mus temporal resolution. These sensors are suited to a diverse range of neutron studies such as microtomography of material composition or phase and strain tomography [2,3,4].
In neutron time-of-flight transmission experiments each neutron arriving at the detector is tagged with its arrival time, this allows the determination of the energy-resolved transmission spectrum from the known time structure of the incident pulse. Variation of these transmission spectra can be used to obtain spatially-resolved maps of projected crystallographic properties within polycrystalline materials; such as average elastic strain, plastic strain, texture and grain size distributions.
Here, we present results from a recent experiment at the Engin-X beamline, ISIS, UK. The transmission spectrum of an additively manufactured jet engine turbine blade was measured and used to determine spatially-resolved maps of the average elastic strain in the transmission direction. This is a sample of intrinsic scientific interest due to questions that remain about the residual strains imparted during fabrication compared to those manufactured conventionally.

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[2] Zhang et al. Mat. Today 2009
[3] Abbey et al. Procedia Engineering 2009
[4] Kirkwood et al. Trans. American Crystallographic Association 2013

Keywords or phrases (comma separated):

Strain mapping, Neutron tomography, Neutron Bragg-edge transmission, Residual strain

Imaging - Sponsored by MASSIVE / 56

Longitudinal Ptychographic Coherent Diffractive Imaging

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In recent years Coherent Diffractive Imaging (CDI) has rapidly matured into a powerful tool for high-resolution X-ray phase contrast imaging. However, a fundamental limit exists on the size of object that can be imaged when using conventional CDI due to the need to correctly sample the measured diffraction intensities. Ptychography, a technique initially developed for electron microscopy,

can overcome limitations on the sample size by combining data collected from multiple overlapping probe positions. Whilst almost all ptychographic CDI experiments are performed using plane-waves our group has been exploring the benefits of introducing phase curvature into the image reconstruction algorithms by illuminating the sample with the diverging probe produced by a focused X-ray beam. We have shown that this geometry allows for rapid image reconstructions from large sample areas with far fewer scanning points needed. Furthermore, by combining data taken with the sample at different longitudinal positions parallel to the incident beam as well as data taken at different transverse positions perpendicular to the beam it has been shown that the dose delivered to the sample can be greatly reduced without loss of spatial resolution. Here we carry this idea further, showing that it is possible to reconstruct an image of a sample scanned through the focal plane without any transverse data being included. This has a number of potential applications for biological imaging including 'zooming in' on regions of interest without imparting potentially damaging X-ray doses to the rest of the sample.

Keywords or phrases (comma separated):

coherent diffraction imaging, ptychography, X-ray imaging

Opening Session / 11

Welcome and Organisational Update

Andrew Peele[None] ; Michael James[1]

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Investigating Molecular Power Converters

Author(s): Daniela Stock[1]

Co-author(s): Alastair Stewart [1] ; Lawrence Lee [1]

[1] *Victor Chang Cardiac Research Institute*

Rotary ATPases are ubiquitous protein complexes that couple the translocation of protons through membranes to the synthesis or hydrolysis of ATP and are thus central to biological energy conversion. Eukaryotic F-type ATP synthases use energy stored in transmembrane proton gradients to synthesise the biological energy carrier ATP from ADP and inorganic phosphate. The evolutionary related V-type ATPases operate in reverse by utilising energy derived from ATP hydrolysis to build up transmembrane ion gradients thereby enabling transport processes across membranes. Most eubacteria have F-type ATPases, but some eubacteria and all known archaea have ATPases of the A-type, which are close homologues of V-ATPases. A-ATPases are simpler in design than their eukaryotic counterparts, but are bifunctional and can operate in either direction in dependence of their cellular environment (1).
We are using a combination of X-ray structure analysis, electron microscopy and other biochemical and biophysical techniques to obtain a pseudo-atomic model of an A-ATPase (2, 3). In addition, X-ray structures in different conformations along with normal mode analysis suggest a greater dynamics of the intact complex than previously envisioned. This might be important for cooperativity and regulation of intact rotary ATPases (4, 5).
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2. Zhou, et al. Science 334, 380-385 (2011)
3. Lee, et al. Nat. Struct. Mol. Biol. 17, 373-378 (2010)
4. Stewart, et al. Nature Communications 3, 687 (2012)
5. Stewart et al. Current Opinion Structural Biology 25, 40-48 (2014)

Keywords or phrases (comma separated):

X-ray crystallography, electron microscopy, bioenergetics, ATP synthase

Opening Session - Sponsored by ANSTO / 13

Determining Molecular Orientation, Packing, and Domain Purity in Organic Photovoltaic Devices with Synchrotron Radiation

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In bulk heterojunction (BHJ) organic photovoltaics (OPVs), electron donating and electron accepting materials form a complex network of discrete and distributed heterointerfaces and charge transport pathways in the photoactive layer where critical photo-physical processes occur. However, little is known about the structural properties of these interfaces due to their 3-dimensional arrangement and the paucity of techniques to measure local order. The presentation will review the use of synchrotron radiation based methods that can uniquely measure critical structural parameters. This includes molecular orientation relative to donor/acceptor heterojunctions [1]. Using resonant soft X-ray scattering [2], the degree of molecular orientation, an order parameter that describes face-on (+1) or edge-on orientation (-1) relative to these heterointerfaces, can be determined. By manipulating the degree of molecular orientation through choice of molecular chemistry and processing solvent characteristics, the importance of this structural parameter on the performance of BHJ OPV devices can be demonstrated. We will furthermore show how compositional variations can be related to polymer crystal size [3] and how mobility and purity can relate to charge extraction and thus in turn to device performance [4,5]. A complete description of actual morphologies and theoretical modeling yet to be developed for OPVs will have to take these factors into account.

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2. B. A. Collins et al., Nat. Mater. 11, 536 (2012).
3. W. Ma at al, Advanced Materials, 10.1002/adma.201400216 (2014)
4. S. Albrecht et al. J. Physical Chemistry Letters 5, 1131-1138 (2014).
5. Collaboration with Wei You and Dieter Neher groups, submitted.

Opening Session - Sponsored by ANSTO / 30

In meso crystallization: Compatibility of Different Lipid Bicontinuous Cubic Mesophases with the Cubic Crystallization Screen in Aqueous Solution.

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A novel in meso crystallization method has facilitated the structural determination of several biologically relevant integral membrane proteins (IMPs). However, the method remains poorly understood as IMPs are difficult to express and handle. Analogous to solution based crystallization, in meso crystallization requires extensive screening of precipitant conditions. Bicontinuous cubic phases are the most commonly used lipid phases for in meso crystallization. The compatibility of the crystallization screen used with the cubic phase is important; if the underlying 3-D cubic nanostructure is destroyed, the screen or protein and lipid combination may not be suitable for in meso crystallization experiments.

We looked at the impact of a screen specifically marketed as compatible with the cubic mesophase, the Cubic crystallization screen (Emerald Biosystems), on the cubic mesophases formed by three different lipids: monoolein, monopalmitolein and phytantriol. The Cubic screen was found to be compatible with cubic mesophase retention under most crystallization conditions studied. The effect of the individual components comprising the multicomponent screen was deconvoluted in two ways. Initially, the effect of specific poly(ethylene glycol) (PEG) and salt components on the cubic mesophase was determined using high-throughput synchrotron Small-Angle X-ray Scattering (SAXS). The effect of high-molecular-weight PEG was shown to dominate the phase behavior within the screen. Finally, a recently developed multiple linear regression modeling method was shown to deconvolute the effect of individual components within the screen effectively.[1]

[1] van 't Hag, L. et al., Crystal Growth & Design, 2014, 14, 1771-1781.

Keywords or phrases (comma separated):

In meso crystallization, High-Throughput Synchrotron SAXS, Cubic crystallization screen

Opening Session - Sponsored by ANSTO / 138

Stimuli Responsive Phospholipid-based Nanomaterials for On-demand Drug Delivery

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Age-related macular degeneration (AMD) is the leading cause of blindness in the elderly, affecting over thirty million people worldwide. The current treatment of wet AMD requires frequent intravitreal injections which are highly invasive and expensive. Therefore, a less invasive and long-lasting treatment is required. One option for achieving such an outcome is using self-assembled lipid-based liquid crystalline (LC) systems, which can encapsulate compounds with varying physicochemical properties and allow delivery of drug to the target site [1]. Drug release from lipid based LC matrices is highly dependent on the nanostructure[2] and has been manipulated to release drug 'on demand' in response to a stimulus, in this case, near-infrared (NIR) light [3]. Such system has potential use in reducing the frequency of injections for short acting or rapidly cleared drugs. Furthermore, the LC nanoparticles can be coated with polyethylene glycol (PEG) for alternative route of administration using the enhanced permeability and retention effect. PEGylation of nanoparticles would prevent non-specific removal from the circulatory system, passively targeting tumour tissues and sites of inflammation, where drug release can then be activated. Thereby, increasing its efficacy as well as reducing the potential for adverse effects. This project is about understanding the phase behaviour of phospholipid systems upon incorporation of PEG-lipids and light-sensitive gold nanoparticles using small angle X-ray scattering to design a stealth and stimuli-responsive LC system suitable for on-demand drug delivery to improve current treatment of AMD.

Opening Session - Sponsored by ANSTO / 139

Pressure-induced coordination change of Ge⁴⁺ and Ga³⁺ in silicate melts

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In geochemical systems, the relative stability of a given element in one phase or another is known as its partitioning behaviour, and many models of Earth processes are based on the changes in partitioning with pressure (and/or other intensive variables). Since higher pressures favour smaller volumes, changes in partitioning with pressure can be predicted if the pressure-dependence of the volume of each phase in the system is known. Volumes of mineral phases can be measured at high pressure, but partial molar volumes of melt components can only be measured at 1 atm. The partial molar volume of a melt component may change with pressure because of the mechanisms of melt compression, such as coordination changes. Si4+ and Al3+, major cations in natural melts, are tetrahedrally coordinated by oxygen at ambient pressure but convert to octahedral coordination at higher pressure. Similar coordination changes may occur for trace cations, yet few have been studied. In this work, x-ray absorption near edge structure (XANES) spectroscopy was used to show a partial coordination change beginning at ~3-4 GPa for trace Ge4+ and Ga3+ in synthetic silicate glasses, quenched from high-pressure melts. Such coordination changes will affect the partitioning behaviour of Ge and Ga between silicate melt and other minerals, and thus have implications for geochemical models such as the depth of formation of erupted magmas and the formation of the Earth's core.

Opening Session - Sponsored by ANSTO / 130

Investigations into the environmental transformations of silver nanoparticles

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Silver nanoparticles (Ag-NPs) constitute a major group of engineered nanomaterials increasingly found in consumer products. These products exploit the unique properties of Ag-NPs such as their antibacterial effects, special optical properties, and high specific surface area and reactivity. However, there are significant concerns regarding the potential for Ag-NPs to pose equally unique risks upon their release to the environment. For example, Ag-NPs may have direct ecotoxicological effects, and they may also release ionic Ag, which is highly toxic to a range of organisms. As AgNP toxicity, dissolution, and speciation are likely to change in response to the surrounding environmental conditions, understanding the transformations of Ag-NPs in major release pathways and environmental endpoints is critical to assessing their potential risks.
To this end, we have developed a series of nano in situ deployment devices (nIDDs) and used them to investigate the transformations of Ag-NPs in a range of environments. Following in situ deployment, during which the Ag-NPs on the nIDDs were directly exposed to relevant environments, the devices were retrieved and X-ray Absorption Spectroscopy (XAS) was used to determine the speciation of the exposed Ag-NPs. This revealed that Ag sulfide NPs are major transformation products forming from Ag-NPs in a wide range of environments. Subsequent XAS-based research showed that Ag sulfide is a stable, long term species of Ag reaching major environmental endpoints such as soil, which mitigates the potential ecological risks posed by the environmental release of Ag-NPs.

Keywords or phrases (comma separated):

Silver nanoparticles, silver sulfide, transformations, fate, nIDDs, XAS

Radiotherapy and Radiobiology / 42

Synchrotron broad beam and MRT radiation induces DNA damage in normal mouse tissues distant from the irradiated volume

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Microbeam radiation therapy (MRT) is a novel, preclinical modality, with a unique ability to generate less radiation damage to neighbouring normal tissues, while providing efficient ablation to the tumour mass; compared to the currently used Broad Beam (BB) modality. A comprehensive investigation on the mechanisms and side effects of these modalities have not currently been established. Here we compared the radiation-induced bystander effect (RIBE) of BB and MRT irradiation, generated by the Imaging and Medical Beamline at the Australian Synchrotron in C57BL/6 mice. Animals were irradiated with 10Gy or 40Gy peak dose of BB or MRT, in an 8x8, 8x1 and 2x2mm area on the right hind leg, using an X-ray beam with a dose rate of 49Gy/sec and constant current of 200mA. At 24 and 96hrs post-irradiation, we collected irradiated skin and an assortment of unirradiated tissue; these were processed for DSB detection, using the γH2AX assay. For both modalities the levels of γH2AX foci in unirradiated tissues of irradiated mice, varied in comparison to irradiated animals. Overall, MRT and BB induced an elevated γH2AX response at 10Gy, while inhibiting this response at 40Gy. Oxidative clustered DNA lesions (OCDL) in tissues were measured using constant field gel electrophoresis, where genomic DNA was treated with purine, pyrimidine and abasic site-specific enzymes. Results show a marked increase of OCDLs in a variety of unirradiated tissues. We will discuss the role of irradiated volume, dose, and beam modality in the manifestation of the in-vivo RIBE.

Keywords or phrases (comma separated):

γH2AX, Microbeam radiation therapy , radiation-induced bystander effect , DNA damage

Radiotherapy and Radiobiology / 44

A high resolution in-beam monitor for microbeam radiotherapy

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Due to the very high dose rates (kGy/s) used in microbeam radiotherapy (MRT), rigorous fluence monitoring is necessary both for pre-treatment verification, and during therapy. We propose an in-beam monitoring system comprised of a 50 μm film of polyethylene terephthalate (PET) metalised with Aluminium positioned in the beam, coupled with a CMOS imaging system for the collection of fluorescence optical photons emitted from the film. We show that such a system has the potential for high spatial and temporal resolution, thereby enabling on-line beam monitoring and treatment verification for MRT.

We position a sample of the film at a 30 degree angle to the beam path in a light tight and shielded enclosure. A CMOS PCO Edge camera with a sensor size of 2560×2160 pixels and a pixel size of 6.5 μm, along with a 105 mm Nikkor macro-lens was nominally positioned at 400 mm to the imaging sensor, and perpendicular to the beam for direct imaging of the fluoresce photons.

The signal to noise ratio for the system was found to be 18.9, and its temporal capabilities were found to be sufficient for detecting beam defining slits in motion, with the MRT beam spectrum at IMBL with a nominal mean energy of 100 keV, and a 1×5 mm field. The proposed imaging system will be a valuable aide in treatment setup, including multi-slit collimator alignment and pre-treatment verification.

Keywords or phrases (comma separated):

beam monitoring, microbeam radiotherapy, optical fluorescence, radiotherapy

Radiotherapy and Radiobiology / 91

Identification of Genes and Molecular Pathways Regulated by Synchrotron Microbeam Radiotherapy

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Synchrotron-generated microbeams radiotherapy (MRT) is a novel preclinical radiotherapy,in which synchrotron-generated X-rays are segmented by a collimator, producing intense microbeams. MRT has been shown to be extremely well tolerated by normal tissues including the central nervous system in animal models when compared to conventional radiotherapy (CRT). The aim of this study was to identify genes and molecular pathways differentially regulated by MRT versus CRT in vitro using cultured EMT6.5 cells. We hypothesized that gene expression and molecular pathway changes after MRT are different from those seen after CRT. We found that at 24 hr post-irradiation, MRT exerts a broader regulatory effect on multiple pathways than CRT. MRT regulated those pathways involved in gene transcription, translation initiation, macromolecule metabolism, oxidoreductase activity and signalling transduction in a different manner compared to CRT. We also found that MRT/CRT alone, or when combined with IFN-ɣ or LPS, up-regulated expression of Ccl2, Ccl5 or Csf2, which are involved in immune cell recruitment. Our findings demonstrated differences in the molecular pathway for MRT versus CRT in the cultured tumour cells. Our findings are consistent with the notion that radiation plays a role in recruiting tumour-associated immune cells to the tumour. Our results also suggest that a combination of MRT/CRT with a treatment targeting CCL2 or CSF2 could repress the tumour-associated immune cell recruitment, delay tumour growth and/or metastasis, and yield better tumour control than radiation alone.

Keywords or phrases (comma separated):

Pathway analysis, in vitro, tumour-associated macrophages, CCl2, CSF2

Radiotherapy and Radiobiology / 59

High Resolution Imaging of IMBL microbeams.

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High resolution 3D imaging of microbeam radiation therapy (MRT) microbeams produced on the Australian Synchrotron's IMBL has been achieved using laser fluorescent confocal microscopy (LFCM). Radiosensitive dosimeters have been specifically fabricated to suit the extremely high dose of the MRT microbeams and the geometrical needs of the LFCM. Cross-fire, stereotactic and interlaced MRT beams are easily resolved in 3D with scope to accurately determine dosimetric properties to be used in future animal and human treatments.

Keywords or phrases (comma separated):

MRT, 3D Microbeam Imaging

Soft Matter / 96

Large soft matter unit cell reconstructions from x-ray and neutron scattering data

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Fourier methods may be used to reconstruct the scattering length density profile of the unit cell from neutron and x-ray diffraction measurements thus yielding information about the distribution of chemical component. Deuteration of the sample components can be used for phasing of the Fourier reconstruction or to provide contrast between components in bilayer stacks1. We discuss the application of selective deuteration of various components of the lipid bilayer and the application of such methods to systems of higher dimensionality such hexagonal and cubic phases and the use of anomalous (energy dependent) diffraction as a means of obtaining phase information.

1. Kent, B.; Hunt, T.; Darwish, T. A.; Hauß, T.; Garvey, C. J.; Bryant, G., Localization of trehalose in partially hydrated DOPC bilayers: insights into cryoprotective mechanisms. Journal of The Royal Society Interface 2014, 11 (95).

Keywords or phrases (comma separated):

neutron diffraction, anomalous x-ray diffraction; medium angle diffraction

Soft Matter / 34

High-throughput synchrotron SAXS studies on lipidic mesophases

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Self-assembled lipidic cubic phases are attracting increasing interest as biocompatible carriers of large biomolecules including proteins, peptides, DNA and drugs [1]. A suite of new high-throughput techniques, to formulate libraries of lipidic mesophases, and structurally characterize them using the SAXS/WAXS beamline at the Australian Synchrotron, are described. Samples are contained within any standard 96-well plate and mounted directly in the beamline. The technique is applicable to any combination of lipids, additives and solvents and sample masses down to 100 μg may be analysed. Up to 8000 samples may be produced robotically and screened in a 24 hr period. The technique is exemplified using the application of membrane protein crystallization [2]. We demonstrate how this high-throughput method allows screening of the extremely large variable physiochemical space for crystallization, which would be unreasonable to explore using traditional experimental methods. In addition the application of this technique to a wide variety of applications for lipidic mesophases will be described, including pharmaceutical (drug discovery, drug delivery, gene therapy and medical imaging), materials science (biosensors, detergent industries), biology (long-term storage of fragile proteins, crystallization) and chemistry/physics (fundamental surfactant and lipid phase behaviour studies), which are currently restricted by difficulties in handling and characterizing this highly viscous material.

1. Conn, C. E.; Drummond, C. J., Soft Matter 2013, 9 (13), 3449-3464.
2. Conn, C. E.; Darmanin, C.; Mulet, X.; Le Cann, S.; Kirby, N.; Drummond, C. J., Soft Matter 2012, 8 (7), 2310 – 2321.

Soft Matter / 53

X-Ray reflectometery on the SAXS/WAXS beamline

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X-ray (XRR) and Neutron Reflectometry (NR) techniques are vital and widely used for characterising the interfacial structure of thin films normal to a surface. XRR is regularly used to characterise systems such as ion distribution at the ionic-liquid/electrode surface, the structure of thin film organic photovoltaics, the structure of organic light emitting devices, phospholipid membranes at the air-liquid interface, etc.
XRR measurement are simple in principle: the intensity of reflected radiation is measured as a function of the momentum transfer, Q, which depends on the angle of incidence and the wavelength of the X-rays. Dividing by the direct beam intensity gives the reflectivity. Providing one has sufficient

incident beam collimation these experiments can be performed on a lab-source x-ray diffractometer/reflectometer.
The energy of most lab sources (typically CuKα) restricts the choice for the upper medium, usually to air. Therefore air/solid or air/liquid interfaces are studied but rarely solid/liquid, liquid/liquid or buried interfaces. XRR at a synchrotron, where the energy can be tuned allied with the high brilliance, enables one to overcome these limitations. We have undertaken experiments at the liquid/solid interface on the SAXS/WAXS beamline using x-ray energies of 20 keV.
This presentation will outline the progress that has been made with the expectation that this technique will be available to the wider user community. The success with organic solar cell dyes studied at the titania/acetonitrile interface will be highlighted.

Keywords or phrases (comma separated):

X-ray Reflectivity, solid/liquid interface

Soft Matter / **54**

SAXS and SANS characterisation of ion irradiation in polymers

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When exposed to swift heavy ion irradiation a wide range of materials show formation of ion tracks as a result of their interaction with the material's electrons. These tracks are narrow, cylindrical-shaped regions of high defect concentration, only a few nanometres in diameter and up to tens of micrometers in length. Ion-irradiated polymers allow the fabrication of microelectronic devices such as micro-capacitors and as well as nanowires, nano-membranes and sensors. We have previously demonstrated small angle x-ray scattering (SAXS) allows a size characterisation of latent tracks in inorganic materials [1].

Here, we present our recent results on the investigation of ion tracks in polycarbonate. SAXS measurements reveal a diameter of 5 nm for tracks hosted within an organic polymer environment. Complementary small angle neutron scattering (SANS) experiments at ANSTO reveal a similar value. However, probing the relative change in density between the latent track and the host material, SAXS shows significant less defect concentration within the tracks than SANS. Both techniques are sensitive to different elements and allow a comparison of the atom-specific damages of ion irradiation in polycarbonate.

Finally, we present the effects of thermal annealing on ion tracks in polycarbonate: Moderate temperatures (100-200 oC) lead to an increase in track diameter, contrary to our previous results on tracks in crystals [2].

[1] P. Kluth et al., Phys. Rev. Lett. 101 (2011) 175503. [2] D. Schauries et al., J. Appl. Cryst. 46 (2013) 1558.

Keywords or phrases (comma separated):

SAXS, ion tracks, ion irradiation, radiation damage, SANS

Structural Biology I / **40**

Human leukocyte antigen-associated drug hypersensitivity.

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Abacavir hypersensitivity syndrome (AHS) is a T-cell mediated drug hypersensitivity triggered by the antiretroviral drug abacavir, used in the treatment of HIV infection. It is one of an increasing number of adverse drug reactions found to be associated with specific Human Leukocyte Antigen (HLA) alleles. Occuring exclusively in individuals possessing HLA-B*57:01, this is one of the strongest associations between an HLA and disease found to date and provides an attractive candidate for exploring the general basis for allotype-specific drug presentation by the HLA. In this study, we detail that abacavir alters the repertoire of endogenous peptides that can bind HLA-B*57:01. By use of the MX2 beamline at the Australian synchrotron we determined the high resolution X-ray crystallographic structure of HLA-B*57:01 in complex with abacavir. This enabled as to map the molecular details underlying the exquisite specificity of abacavir for HLA-B*57:01. Furthermore, we were able to show that abacavir changes the shape and chemistry of the HLA-B*57:01 antigen-binding cleft. In this way, abacavir guides the selection of new endogenous peptides, inducing a marked alteration in 'immunological self'. The resultant peptide-centric 'altered self' activates abacavir-specific T-cells, thereby driving polyclonal CD8+ T-cell activation and a systemic reaction manifesting as AHS.

Reference: Illing, P., Vivian., et al
Nature. 2012 Jun 28;486(7404):554-8
Immune self-reactivity triggered by drug-modified HLA-peptide repertoire.

Keywords or phrases (comma separated):

Immunology, Adverse drug reactions, Human Leukocyte antigen

Structural Biology I / 38

Structural Insights into Bak Activation and Oligomerisation

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Apoptotic stimuli activate and oligomerise the pro-apoptotic proteins Bak and Bax resulting in mitochondrial outer membrane permeabilisation and subsequent cell death. Crystal structures by Czabotar et al. (2013) provided novel insights into BH3-only induced Bax activation and oligomerisation, namely the separation of the core and latch domains, followed by core domain dimerisation. Here we provide complementary studies on the related protein Bak. We present the crystal structures of Bak core-latch domain swapped dimers and demonstrate their dissociation upon Bak activation. A second crystal structure of the Bak core domain provides the first high-resolution details for this key dimerisation unit upon which the larger Bak oligomer builds. Cellular assays, guided by the presented crystal structures, confirm the physiological relevance of these key events in the intrinsic apoptotic pathway (Brouwer et al. 2014). These studies confirmed an analogous mechanism for activation and dimerization of Bak and Bax in response to BH3-only peptides. More recently we have performed structural studies on the direct interaction of BH3-only peptides with Bak. We have gained insight into the differences between interactions of BH3 only proteins with Bak compared

to the pro-survival proteins; this may inform the design of novel therapeutics to manipulate cell death.

References

Brouwer et al. (2014), Mol Cell, published online 28th August, http://dx.doi.org/10.1016/j.molcel.2014.07.016

Czabotar et al. (2013), Cell 152(3): 519-531.

Keywords or phrases (comma separated):

Apoptosis, Bak, Activation, Oligomerisation

Structural Biology I / 82

Structure and function studies on human plasminogen glycoforms

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Plasminogen is a 7-domain protein (with an N-terminal Pan-apple domain, five kringle domains and a C-terminal serine protease domain) that adopts a closed, activation-resistant conformation in the circulatory system. The recruitment of plasminogen to its target sites is dependant on the lysine binding sites of the kringle domains. Binding to lysine residues on cell receptors and fibrin clots simultaneously triggers a conformational re-arrangement of the molecule to adopt an open conformation. The open form is readily converted to plasmin by tissue- and urokinase-type Plasminogen Activators. Plasmin plays a key role in number of physiological and pathological processes including degradation of extracellular matrices, cell migration, tissue remodeling, wound healing, angiogenesis, inflammation, pathogen invasion and cancer migration.

We have solved the X-ray crystal structure of human plasminogen in the closed conformation. Our results revealed that the N-terminal Pan-apple domain and the serine protease domain maintain the closed conformation via interactions made throughout the kringle array, in particular, Kringles 2, 4 and 5. Our data suggests that Kringle 1 governs proenzyme recruitment to target sites and binding to external lysine of Kringle 5 in the closed conformation may trigger the formation of the open conformation.

Glycosylation affects the overall conformation of the protein and therefore its functions. There are two plasminogen glycoforms in the plasma; our structural data suggests that these glycoforms have distinct structural characteristics. Here we discuss our studies on the activation and inhibition of these two glycoforms.

Keywords or phrases (comma separated):

Glycosylation, fibrinolysis, conformation, crystallography, SAXS

Structural Biology I / 8

Winning the battle of Signal vs Noise

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The success or failure of any structure determination effort is dictated by the signal-to-noise ratio, so a quantitative understanding of both signal and noise is needed to have the best chance of success and to avoid wasted effort on samples that simply aren't good enough. There are three main hurdles to every structure determination effort: the Phase Problem, the Amplitude Problem, and the R-factor Gap. The Phase Problem is hampered mainly by sources of relative error, such as shutter jitter, incident beam flicker, sample vibration, detector calibration, and non-isomorphism, including that induced by radiation damage. High multiplicity is the best way to combat relative error, but it must be "true" multiplicity, where no spot is ever measured the same way twice. Conversely, the Amplitude Problem, also known as "poor diffraction" is dominated by the background under weak spots, and the finite number of photons appearing in a weak spot before the crystal dies. This radiation damage limit can be outrun with femtosecond pulses, but even X-ray Free Electron Lasers (XFELs) cannot make a disordered crystal diffract. Screening for order is best performed at synchrotrons. The R-factor Gap is the large discrepancy between (Rcryst/Rfree) and the error in the data itself (Rmerge/Rmeas). Closing this Gap requires a better understanding of dynamics, which itself is relevant to function. Overall, the Three Hurdles can be overcome by improved methodology, better sample preparation, and improved macromolecular models that will open new doors in structural biology.

Structural Biology II / 76

A Two-pronged Attack: Dual Inhibition of Plasmodium falciparum M1 and M17 Metalloaminopeptidases by a Novel Series of Hydroxamic acid-based Inhibitors.

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Malaria is caused by parasites of the genus Plasmodium, with Plasmodium falciparum (Pf) causing the most deaths. The prevention and treatment of Pf malaria is becoming increasingly difficult due to the spread of drug resistant parasites. New therapeutics with a novel mode of action are desperately required. Two Plasmodium falciparum aminopeptidases, PfA-M1 and PfA-M17, play crucial roles in the erythrocytic stage of infection, and have been validated as potential antimalarial targets. Using compound-bound crystal structures of both enzymes, we were able to identify key similarities and differences in the mechanism of inhibitor binding by PfA-M1 versus PfA-M17, which we exploited to design inhibitors capable of potently inhibiting both enzymes. The resultant hydroxamic acid-based inhibitors represent the first compounds capable of potent dual inhibition of both PfA-M1 and PfA-M17. The compounds additionally possess nanomolar activity against 3D7 malaria parasites and no observable cytotoxicity, and are therefore extremely attractive lead molecules for further development into antimalarial therapeutics with a novel mode of action.

Keywords or phrases (comma separated):

falciparum malaria, aminopeptidase, inhibitors, hydroxamic acid, structure-based drug discovery

Structural Biology II / 77

Structural Studies of Alzheimer's Disease Amyloid Precursor Protein Dimers

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Amyloid precursor protein (APP) is a type-I transmembrane protein with a large ectodomain (sAPP), a single transmembrane domain and a cytoplasmic tail. It is cleaved by beta- and gamma-secretases to generate amyloid-β (Aβ), a neurotoxic peptide implicated in Alzheimer's disease (AD). APP dimerisation is closely linked to Aβ overproduction. It is also implicated in APP signalling as APP is proposed to be membrane receptor. There are four potential dimerisation sites in APP, three of which are located in the sAPP region. However, the mechanism of APP dimerisation remains unclear. Understanding APP dimerisation mechanisms at the molecular level will not only provide insights into how APP signals but also have therapeutic implications. Several factors are found to regulate APP proteolysis in vivo. To test their impact on sAPP dimerisation, we used in vitro techniques including ThermoFluor, analytical ultracentrifugation and multi-angle light scattering. We found certain metals and sugars synergistically drive sAPP dimerisation, which is associated with conformational changes and increased stability. This discovery has led to the successful crystallisation of sAPP in its dimeric forms. The identities of metal ions were confirmed using anomalous difference Fourier maps. Key features underlying sAPP dimer formation are clearly demonstrated in the structure. Together with results from the solution studies, the crystal structure has provided a leap forward in understanding the mechanism of APP dimerisation and its pathogenic and functional implications. The structure will be used to discover inhibitors of APP dimerisation and Aβ overproduction as possible AD therapeutics.

Keywords or phrases (comma separated):

Alzheimer's, APP, dimerisation, metal binding, crystal structure

Structural Biology II / 35

Structural insights into the organization of the cavin membrane coat

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Caveola membrane invaginations are a striking feature of many vertebrate cell types, and are critical for cell signaling, endocytosis and mechanotransduction. Their formation depends on the caveolins and the cavin peripheral membrane proteins (cavin1, cavin2, cavin3 and cavin4), although there is currently no atomic level information addressing the mechanisms that underpin caveola assembly. Here we show that a minimal N-terminal domain of the cavin proteins (the HR1 fragment) is required and sufficient for their homo and hetero-oligomerisation. The crystal structures of mouse cavin1 and zebrafish cavin4 HR1 domains reveal highly conserved trimeric coiled-coil architectures, with unique intra-subunit interactions that determine the specificity of coiled-coil formation. A conspicuous feature of the HR1 domain is a basic surface patch, conserved among all cavins and across all species, which we show can mediate interaction with negatively-charged membrane lipids including phosphoinositides. Mutations in this domain prevent membrane association and perturb

caveolae formation in vivo. Interestingly the cavin proteins possess intrinsic membrane remodeling properties in vitro, that we propose is important for the formation of caveolae. Finally, we show that full-length cavin proteins possess characteristic rod-shape structures that reflect the coiled-coil architecture of the HR1 assembly domain and have dimensions corresponding closely to the striations observed on the surface of caveolae in vivo. We therefore propose the striations forming the common coat of caveola are composed of polymerised cavin trimers.

Keywords or phrases (comma separated):

Protein crystallography, coiled-coil, membrane trafficking

Structural Biology II / 115

Crystal Structure of Human Insulin-Regulated Aminopeptidase

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Dementia is the single greatest cause of disability in older Australians afflicting almost one in ten over the age of 65. In the absence of curative therapies, current treatments aimed at enhancing working memory target the cholinergic system and demonstrate limited efficacy, underpinning the need for a new class of cognitive enhancing drug. Insulin-regulated aminopeptidase (IRAP) is a membrane-bound zinc-metallopeptidase that cleaves neuroactive peptides in the brain and its inhibition gives rise to memory enhancing effects in both normal and memory-impaired rodents . Using a large scale insect cell expression system to produce milligram quantities of protein suitable for crystallography, and the Micro Crystallography Beamline at the Australian Synchrotron, we have determined the crystal structure of human IRAP to 2.96 Å. This structure revealed a semi-closed, four domain arrangement with a large, mostly buried cavity adjacent to the active site as well as a dimer interface located in the C-terminal domain. A comparison of the catalytic domain with related aminopeptidases revealed a strikingly different conformation of the GAMEN exopeptidase loop that explains IRAP's unique specificity for cyclic peptides such as oxytocin and vasopressin. This structure will be a powerful tool in the development of new classes of cognitive enhancers for treating memory disorders such as Alzheimer's dementia.

Keywords or phrases (comma separated):

Alzheimer's disease, Memory enhancing, Crystallography, Aminopeptidase

Surface Science / 70

Probing Film Morphology and Surface Microstructure of Semiconducting Polymers with GIWAXS and NEXAFS

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In making organic electronics a reality, donor-acceptor based semiconducting polymers will play a pivotal role. The molecular packing, orientation and crystallinity of semiconducting polymer thin-films strongly influence the performance of organic electronic devices. Grazing Incidence Wide-Angle X-ray Scattering (GIWAXS) collected at the SAXS/WAXS beamline has been used to probe the molecular packing and relative crystallinity of polymer thin films; while Near Edge X-ray Absorption Fine Structure (NEXAFS) spectroscopy at the soft X-ray beamline has been used to probe the microstructure and molecular orientation of film-surfaces. A novel polymer BFS4 is studied, which is based on a dithienyl-benzo[1,2-b':4,5-b']dithiophene as a donor-unit and 5-fluoro-2,1,3-benzothiadiazole as an acceptor-unit. GIWAXS reveals that the crystallites are in a mixture of orientations where the in-plane alkyl-chain stacking distance is 2.17 ± 0.01 nm and out-of-plane π-stacking distance is 0.385 ± 0.005 nm. Coherence lengths are measured to be 15 ± 2 nm and 2.6 ± 0.2 nm respectively. Two different types of solution processing methods show differing crystallinity, which can be correlated directly to their respective transistor charge transport properties. NEXAFS reveals a mixture of edge-on and face-on orientations of the molecular planes, and the same orientational preference is confirmed. NEXAFS additionally reveals the relative orientation of the donor and acceptor units of the backbone of this novel polymer, probing backbone planarity directly.

Keywords or phrases (comma separated):

Organic electronics, Semiconducting polymers, Film Morphology, Surface Microstructure, Molecular orientation

Surface Science / 64

Morphological investigations of naphthalene diimide derivatives

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Solution-cast, organic field-effect transistors (OFET) have many advantages, such as rapid, large area fabrication, low production cost and flexible substrates making them ideal for specialized application such as flexible displays and radio frequency identification.
The small molecule organic semiconductor (OSC) naphthalene diimide (NDI) provides a versatile framework with which to build upon and explore the effects of chemical functionalisation.
Over the past year, we have utilized two complimentary synchrotron based techniques to examine the effect on structure and morphology as the basic NDI framework is functionalised.
At the soft x-ray beamline, Near Edge X-ray Absorption Fine Structure (NEXAFS) spectroscopy allowed us to explore the surface molecular orientation and electronic changes. Expansion of the NDI core was found to give higher average tilt angles with core-expanded derivatives also maintaining their high average tilt angle when annealed.
At the SAXS/WAXS beamline these same materials were investigated using Grazing Incidence Wide Angle X-ray Scattering (GIWAXS) to assess crystallinity and molecular packing. The data confirms both the highly edge on packing behaviour of the core expanded set and the drastic change to the molecular unit cell upon NDI core expansion.The $\pi - \pi$ stacking distance of the conjugated cores, which is an important element of transistor design to allow effective charge transfer, is also measured for each of the materials.
This information will aid and inform the ongoing design of our NDI molecules as we progress towards a high mobility, air stable OFET.

Keywords or phrases (comma separated):

Small molecule, organic semiconductor, OFET, n-type

Surface Science / 80

Characterisation of scale products formed on the heat-exchanger surfaces in the Bayer process

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Vast quantities of bauxite ore, used for the production of alumina (Al2O3) via the Bayer process, contain appreciable concentrations of reactive silicates. Secondary precipitation of these silicates within Bayer plants results in deleterious scale formation consisting largely of aluminosilicate and titanate phases, with resulting losses of caustic soda and decreased heat transfer efficiency. In spite of numerous related laboratory studies, an insufficient amount of data has been published regarding the characterisation of these industrial scales, which can provide vital information pertaining to the mechanism of scale growth. SEM, XRD and EDS analyses were carried out on selected scale samples removed from the inside wall of plant equipment across a number of alumina refineries. SEM images of the cross-section of the industrial scale samples were carried out to explore compositional changes with depth from the wall. High amorphous content of the industrial samples were calculated using XRD Rietveld analysis and the addition of corundum was used as an internal standard. Therefore, XANES at both the Al and Si K-edges was applied to identify the amorphous phases, in conjunction with XRD and EDS results, which were demonstrated to consist predominantly of poorly crystallised sodalite. There is strong correlation between the amorphous content and the scale microstructure. High amorphous content appears to indicate nucleation of the scale occurred on the internal surfaces of the heat exchangers. Low amorphous content suggest the desilication product particles form in the slurry during desilication and then deposit on the heat exchange surfaces.

Keywords or phrases (comma separated):

Bayer process, XANES, scale, characterisation

Surface Science / 29

Stability and Surface Reconstruction of Bi2Se3 on Exposure to Atmosphere

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The new class of topological materials including Bi2Se3 offer opportunities to develop next generation electron devices that utilize spin generation and detection without ferromagnetism [1]. However, the fate of the Bi2Se3 surface upon exposure to atmosphere remains unclear. In particular whilst the topology of Bi2Se3 guarantees the presence of a metallic surface, the topological properties of the metallic surface depend on the surface and its reconstruction [2]. Therefore, it is essential to understand the structure of the air-exposed Bi2Se3 surface in order to interpret the properties of any air-exposed Bi2Se3 device.

Utilizing high-resolution surface sensitive XPS we reveal that five minute air exposure causes a drastic change to the surface of in-situ cleaved Bi2Se3. An additional component within the Bi 5d core level was observed after exposure that corresponds to the formation of isolated ~0.8 nm thick

Bi2 layers at the surface of Bi2Se3 [3]. This Bi2 layer is found to occur across multiple samples and is precipitated rapidly by exposure to atmosphere, while samples left for several days in UHV after in-situ cleaving show no change. This finding offers new avenues to study a 2D TI (Bi2) interfaced with a 3D TI (Bi2Se3) but also has significant consequences in understanding the electronic structure of air-exposed Bi2Se3.

[1] Y. Xia, et al., Nature Physics 5, 398 (2009).
[2] Q. D. Gibson, et al., Phys. Rev. B 88, 081108(R) (2013).
[3] M. T. Edmonds, et al., J. Phys. Chem. C 118, 20413 (2014).

Keywords or phrases (comma separated):

Bi2Se3, Surface Reconstruction, Topological Insulator

Welcome Function, Poster Session, Exhibition - Board: 802 / **47**

The effect of increasing radiation doses on normal & malignant cell migration

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The aim of the project is to investigate how normal and malignant cell migration is influenced by conventional radiotherapy doses as well as by experimental treatments such as microbeam radiation therapy (MRT). Radiation-induced tumor cell migration is a recognized phenomenon that can occur when cells are sub-lethally irradiated. Our group previously demonstrated that tumor cells showed extensive migration 24 hours post-MRT.
We irradiated well chamber slides containing cultured normal and malignant cells with a range of doses (2, 5, 10Gy) using a
conventional, Cobalt- 60 source. We used time-lapse microscopy (live cell imaging) and image processing techniques to track fluorescently-labelled cells for up to 24 hrs post-RT.
Statistical analysis of the live cell microscopy data showed there was a notable change in the vector displacement when cells were irradiated with increasing doses of conventional radiation. The observed changes in cell movement may be due to asynchronous cell cycling and proliferation, subsequently dampened with increasing dosages of radiation. We noted a bi-phasic response in some cases with the initial 'hit' of radiation generating an immediate response which switches over to a longer-term response when the damage finally takes its toll on the cells and they slow
down to die.
The cells' migratory capacity was clearly affected or modulated by the conventional
radiation doses. This modulation could be important for MRT studies since the low, 'valley' dose may leave cells sub-lethally irradiated.

Keywords or phrases (comma separated):

microbeam radiation therapy, cell migration

Welcome Function, Poster Session, Exhibition - Board: 1009 / **41**

Structure characterization of the Chlamydomonas reinhardtii magnesium chelatase GUN4 and H subunits by small-angle X-ray scattering

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The magnesium chelatase enzyme catalyses the ATP dependent insertion of Mg+ in to protoporphyrin IX(PPIX) in the first step of the chlorophyll biosynthesis pathway consists three different protein subunits ChlI ChlD and ChlH. The GUN4 protein is a regulatory subunit of Mg-chelatase that binds the chlorophyll biosynthesis intermediates, PPIX and Mg protoporphyrin(Mg-PPIX), stimulates Mg chelatase activity, and is implicated in developmental signaling pathway between the chloroplast and nucleus. ChlH is the largest subunit of Mg-chelatase which also binds both PPIX substrate and the Mg-PPIX product. GUN4 appears to participate in a plastid-to-nucleus signalling pathway possibly through regulating Mg-PPIX synthesis or trafficking. Unlike the cyanobacterial GUN4, the chloroplastic orthologous have an extra C-terminal domain that is phosphorylated and is required for magnesium chelatase activity. We have determined the low resolution solution structure of GUN4 , H and the GUN4-H-PPIX complex at ~20 A°, by using (SAXS) small-angle x-ray scattering and can report that the GUN4 protein has a more elongated structure compared to the cyanobacterial protein. Furthermore, The SAXS structure of the GUN4-H-PPIX complex is similar to the SAXS structure of H subunit suggesting that GUN4–PPIX may attach somewhere inside the cage shape structure of H subunit to form a complex.

Keywords or phrases (comma separated):

Mg-chelatase, GUN4, ChlH

Welcome Function, Poster Session, Exhibition - Board: 103 / **71**

Correlating morphology and device physics of high open circuit voltage, low-band gap all polymer solar cell using various characterization tools.

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The microstructure and device physics of photovoltaic polymer blends based on the donor polymer BFS4 (a dithienyl-benzo[1,2- b:4,5-b]dithiophene / 5-fluoro-2,1,3-benzothiadiazole co-polymer) paired with the naphthalene diimide-based acceptor polymer P(NDI2OD-T2) will be presented. Efficiencies of over 4% are demonstrated, with an open circuit voltage of greater than 0.9 V achieved. Near-edge x-ray absorption fine-structure (NEXAFS) spectroscopy and atomic force microscopy (AFM) measurements reveal that the top surface of BFS4:P(NDI2OD-T2) blends is covered with a pure BFS4 capping layer. XPS Depth profiling measurements confirm this vertical phase separation with a surface-directed spinodal decomposition wave observed. Grazing-incidence wide-angle x-ray scattering (GIWAXS) measurements confirm that BFS4 and P(NDI2OD-T2) are semicrystalline with both polymers retaining their semicrystalline nature when blended. Transmission electron microscopy reveals a relatively coarse phase-separated morphology, with elongated domains up to 200

nm in width. Photoluminescence spectroscopy reveals incomplete photoluminescence quenching with as much as 30% of excitons failing to reach a donor/acceptor interface Addition of DIO as solvent improves the fill factor of the devices from 0.46 to 0.54, thus improving the overall efficiency from 3.9% to 4.5%. Effect of addition of DIO in the neat polymers and blends is also studied using NEXAFS and GIWAXS techniques. NEXAFS and GIWAXS measurements were performed at the Australian Synchrotron, Soft X-ray and SAXS/WAXS beamlines respectively.

Keywords or phrases (comma separated):

OPV, Morphology, GIWAXS, NEXAFS

Welcome Function, Poster Session, Exhibition - Board: 1011 / **73**

Targeting DsbD from Neisseria meningitidis for the development of new anti-Neisserial agents

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The lack of antibiotic development coupled with the rapid increase of resistance to antibiotics in bacteria, has led to a situation described as an 'alarming public health crisis'(1). Multi-drug resistant (MDR) bacteria are becoming a significant problem because some bacterial strains cannot be treated with our current strongest and last resort antibiotics. There is an urgent need to develop alternative strategies to combat bacterial infections. Two MDR bacteria are Neisseria meningitidis, the causative agent of meningitis, and Neisseria gonorrhoeae, the causative agent of gonorrhoea. The overall aim of this work is to develop a narrow spectrum antibiotic against N. meningitidis. The biological target: NmDsbD, is a disulfide bond (Dsb) reductase that is required for the viability of N. meningitidis (2). NmDsbD is membrane bound and consists of three redox active domains: two are periplasmic domains, n-NmDsbD and c-NmDsbD, which flank the transmembrane domain, t-NmDsbD. In this work we solved the crystal structures of n-NmDsbD and c-NmDsbD, and have briefly investigated the interaction between these two domains. A fragment-based drug design approach was also used to identify small molecules that bind both n-NmDsbD and c-NmDsbD.

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2. Kumar, P., et al. (2011) Characterization of DsbD in Neisseria meningitidis. Molecular microbiology 79, 1557-1573

Keywords or phrases (comma separated):

DsbD, crystal structures, Fragment-Based Drug Design, Neisseria meningitidis

Welcome Function, Poster Session, Exhibition - Board: 105 / **74**

A New and Novel Approach to the Formation of Metal-Metal Bonded Complexes using "Inorganic Grignard Reagents"

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The synthesis, structure, bonding and reactivity of molecular compounds containing unusual metal-metal bonds continues to be a topic of considerable interest. We have added to this field by utilising extremely bulky monodentate amido ligands for the stabilisation of a number of low oxidation state transition metal complexes, of which some have shown reactivity comparable to that of the classical Grignard Reagent.

These complexes were initially prepared from the reduction of amido metal halide precursors (LMX) (L = bulky amido ligand; M = transition metal, X = a halide) with the novel magnesium(I) reducing agent [{(MesNacnac)Mg}2]. Using bulky ligands, this reduction step often yields the formation of a low coordinate transition metal(I) dimer (LMML). However, by using extremely bulky ligands, the formation of the dimer is unfavourable and a number of novel LM-Mg(MesNacnac) heterobimetallic complexes can instead be isolated. These unprecedented complexes contain the first examples of a number of metal-metal bonds, such as manganese-magnesium and zinc-magnesium.

The metal-magnesium bonded compounds have shown to act as "inorganic Grignard Reagents" in reactions with a number of metal(I) and metal(II) halide complexes, in which their LM fragments are transferred onto other metal centres. This novel reactivity is still being investigated but has already allowed access to number of previously inaccessible hetero-bimetallic and even trimetallic complexes. All aforementioned complexes were structurally characterised by X-ray crystallography using the Australian Synchrotron MX beamlines.

Keywords or phrases (comma separated):

chemistry, inorganic, x-ray, crystallography, MX, transition, metal, grignard,

Welcome Function, Poster Session, Exhibition - Board: 111 / **75**

Enantionmeric Separation Using Entangled Coordination Polymers

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Chiral coordination polymers, using ligands that contain large aromatic cores have been used to synthesize a series of polycatenanes and a polyrotaxane, by virtue of π interactions involving metallomacrocyclic motifs, including interpenetrated networks that have been used as stationary phases to obtain excellent enantiomeric resolution in liquid-chromatographic separations.
A series of dicarboxylate ligands, in which amino acids are appended to naphthalene- or perylene-diimides, have been used to readily impart chirality into coordination polymers. A repeating structural motif, in which dinuclear metal clusters are bridged by two ligands to form a metallomacrocycle with a separation of ca. 7 Å between opposing aromatic faces – an ideal distance for hosting an aromatic guest. This motif has been exploited to form polycatenanes, through self-complementary association of the metallomacrocycles, and a 1D⟶3D polyrotaxane when using a 4,4'-bipyridyl co-ligand as a linear thread.
Two 2D⟶3D interpenetrated materials have been shown to act as efficient stationary phases for enantioselective separations in small-scale testing with 1-phenylethanol, pantolactone and limonene. The separation activity of the material is much greater than that of the free ligand alone.

Keywords or phrases (comma separated):

Coordination Polymers, Crystallography

Welcome Function, Poster Session, Exhibition - Board: 1012 / **149**

Characterisation of model protein denaturation by SAXS

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The biological functions of proteins depend on the ability of the proteins to fold correctly. Misfolding/unfolding of proteins can cause formation of insoluble pathological aggregates, leading to degenerative diseases, such as Alzheimer's and Parkinson's [1]. Although considerable studies have been carried out on the molecular mechanism of protein aggregation, the lack of detailed information that links the initial stage of aggregation and the final structure limits the understanding of protein aggregation. In this study, our objective is to investigate the thermal denaturation and pre-aggregation of three soluble model proteins, ribonuclease A, myoglobin and chymotrypsinogen A, with different secondary structure characteristics. By using in situ small angle X-ray scattering, we can extract thermodynamic parameters and study the very early stages of the aggregation process-especially the conformational changes of individual protein molecules in solution on thermal denaturation. Our hypothesis is that the differences in aggregation mechanisms observed in the models proteins during thermal denaturation are intimately related to their secondary structures. These results will be complemented with chemical and high pressure denaturation to gain a complete thermodynamic picture of the different protein-types resistance to aggregation.

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Keywords or phrases (comma separated):

Protein aggregation, thermal denaturation

Welcome Function, Poster Session, Exhibition - Board: 210 / **148**

Extreme environments at the XAS beamline

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X-ray Absorption Spectroscopy is a technique for structure determination and speciation studies which is well suited to the in situ study extreme environments. Extreme environments are systems under high pressure and high temperature. These systems occur in nature when geofluids dissolve metals from large regions of host rock, and later precipitate the metals as ore deposits. Apart from understanding the formation of ore deposits, understanding geofluids is important in developing ore processing, and for geothermal power stations.

Two new systems for studying extreme environments are being implemented in hutch C at the XAS beamline. Hutch C is a separate experimental station to hutch B, the main experimental hutch where routine XAS experiments re performed. One of the aims of the extreme environments project is the set up hutch C to run independently of hutch B. An example is the parallel implementation of motor control, and detector counting chains.
The first new system is the maestro autoclave cell, developed by Joel Brugger et al. The maestro cell is a large volume cell which can run temperatures from 25 – 600 degrees C, and pressures from 1 to 600 bars. The large volume cell allows us to study dilute metal ions in solution such as geofliuds encountered in nature.

The second system is the Hydrothermal Diamond Anvil Cell (HyDAC). This cell can impart pressures between 0.1 – 5 GPa, and temperatures 25 – 1000 degrees C. The volume of the HyDAC is made larger than other DACs by milling into the two diamonds.

Keywords or phrases (comma separated):

XAS, XAFS, XANES, Extreme environments, geofluids

Welcome Function, Poster Session, Exhibition - Board: 209 / **143**

Opportunities for a greater spectroscopy DCM - vibration study

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X-ray absorption spectroscopy (XAS) is a technique that typically challenges the performance of its monochromator. The XAS Beamline at the Australian Synchrotron utilises a commercially delivered liquid Nitrogen cooled double crystal monochromator (DCM). This DCM possesses excellent energy and beam offset stability. Some mechanical and electrical improvements have been made in-house to enhance performance for the beamline's science user community. Further improvements to the DCM are strongly motivated by the research needs of the user community, for eg materials and systems where a metal species of interest is present at very low concentrations (few 100 ppb).

For any DCM data quality can be limited by vibrations between the two diffracting crystal surfaces, resulting in beam 'noise' via positional changes, flux intensity perturbations and possibly small energy changes. As such, effort is invested to reduce current DCM crystal vibrations (~400-1000 nRad) to increase system performance and satisfy user community needs.

Herein we report an in-situ (with X-ray beam and a position sensitive ion chamber) and ex-situ (with accelerometers) characterisation of the vibrational response of the DCM at the XAS Beamline. In general, the X-ray beam shows a vibration spectrum closely resembling the measured resonant frequencies of the DCM components, as well as the superposition of the floor dynamics. The effect of rubber damping pads, stiffened supporting structures and other modifications will be presented, and ideas and strategies for improved vibrational performance will be discussed.

Keywords or phrases (comma separated):

XAS, monochromator, beamline, spectroscopy

Welcome Function, Poster Session, Exhibition - Board: 605 / **144**

X-ray Micro-Tomography using fluorescence from a metal foil as a light source combined with X-ray Micro-Diffraction

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Micro-X-ray computed tomography is a well established 3D imaging method that plays an important role in fields like materials science, food research, and bio-medical imaging. It is interesting to

combine tomography with other techniques, such as X-ray diffraction, for assessing physical properties (density and crystallography) of materials that are heterogeneous on the micrometer scale. The challenge lies in reconciling the needs of both methods, whereby tomography typically requires a large beam and micro-diffraction requires a small beam.

To bridge this gap, a study was conducted at the MX1 beamline. Tomographic imaging was realised by placing a metal foil upstream from the sample thus generating a diverging fluorescence wavefront (1). This wavefront was propagated through the sample onto the beamline's CCD detector for absorption contrast imaging. The relatively small beam (~150mu) was used for simultaneous small-scale illumination for micro-diffraction.

To explore some of the possibilities of the combined method, imaging results from different density materials will be presented. Parallel micro-diffraction is demonstrated using an Egyptian faience bead, and elemental contrast imaging is explored by imaging Ni and Cu metal meshes. This study highlights possibilities to obtain three-dimensional structural information simultaneously on micrometer and crystallographic length-scales. The results show promise for further developments in element contrast imaging, and results from the work also inform possible extensions to phase contrast imaging.
(1) P.Kappen, B.Arhatari, M.B.Luu, E.Balaur, and T.Caradoc-Davies, Rev. Sci. Instrum. 84, 063703 (2013).

Keywords or phrases (comma separated):

micro-tomography, micro-diffraction, X-ray fluorescence, MX1 beamline

Welcome Function, Poster Session, Exhibition - Board: 307 / **147**

SAXS as an assay for protein:ligand interaction.

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Small-angle x-ray scattering (SAXS) is sensitive to the size and shape of macromolecules in solution. Whilst there can be some uncertainties involved in modelling macromolecular structure directly from SAXS data, relative changes in structure can be measured with a high degree of confidence.

The binding of a small-molecule ligand to a large protein in itself is generally too subtle an event to see with confidence by SAXS. In order to use SAXS as an assay for the interaction between a ligand and a protein one of the following cases must be true: 1) the ligand interaction causes a conformational change in the protein, 2) the ligand interaction disrupts a protein:protein interaction causing a change in multimerisation state or 3) the ligand itself is rather large by comparison to the protein.

On this basis I present three recent examples that explore the utility of SAXS to assaying the interaction between medically relevant proteins and various chemical ligands. These examples are: 1) the binding of lysine analogues with the human anti-clotting protein plasminogen that lead to a dramatic conformational change in the protein, 2) the interaction between a large branched polymer (dendrimer) and HIV gp120 that leads to a reduction in infection rates and 3) interaction between a novel allosteric inhibitor and HIV reverse-transcriptase that causes a subtle rearrangement in the domain structure of this protein and lead to inhibition of function.

Keywords or phrases (comma separated):

protein, structure, kinetics, SAXS

Welcome Function, Poster Session, Exhibition - Board: 609 / **146**

High-Definition X-ray Fluorescence Elemental Mapping of a Early Tudor Portrait of Henry VIII

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[2] *Art Gallery of New South Wales*

A Tudor portrait of Henry VIII on oak dating from 1540s is currently undergoing conservation treatment. This treatment includes the removal of restoration paint layers applied prior to its acquisition by the Art Gallery of New South Wales. Conventional imaging techniques, x-radiography, infrared reflectography and ultraviolet fluorescence suggested damages underneath the restorations. These include a break in the panel through the centre, the addition of an extra panel piece along the right edge and extensive overpaint by previous restorers. The addition of balsa wood strips to the verso of the panel using a wax and chalk adhesive caused significant interference in the conventional x-radiograph image.

High-definition XRF elemental mapping of the painting produced images of the distribution of original and non-original paint layers across the painting. Original and non-original paints were distinguished through historical studies of known pigments from the 16th century such as bone black, natural chalk vermillion, copper-based greens and blues, gold leaf, lead white and iron-based earth colours against those manufactured later, such as chrome greens.

The elemental maps are proving invaluable guides in the removal of restoration materials and the retrieval of original details previously obscured, and is providing unique insight into the making of the painting and suggests that miniature and manuscript illumination may have been important technical sources for the artist.

Keywords or phrases (comma separated):

Paintings XRF elemental mapping imaging cultural heritage

Welcome Function, Poster Session, Exhibition - Board: 101 / **5**

New Formamidinate Rare Earth Metal Complexes by Pseudo-Grignard Reaction

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Pseudo-Grignard reagents1, 2, ‘‘RLnX’’ (Ln = Eu, Sm and Yb; R = Me, Ph or C6H2Me3-2, 4, 6; X = Br, I), formed by the treatment of organic halides like PhBr or PhI with rare earth metals in Lewis base solvents, can be employed to various organic or inorganic transformations3, 4. We now report the synthesis of new divalent rare earth metal formamidinate complex [Ln(Form)Br(thf)2]2 through the relevant Pseudo-Grignard reactions of rare earth metal with bromobenzene in the presence of formamidine (Eq. 1).

A typical reaction using ytterbium metal and bromobenzene or iodobenzene in the presence of 2,4,6-methylformamidine led to the isolation of orange and light orange complexes [Yb(Form)Br(thf)2]2, [Yb(Form)I(thf)2]2. The product has been structurally characterized including X-ray crystallography (Fig. 1).

Fig. 1. Molecular structure of [Yb(Form)Br(thf)2]2

Fig. 2. Molecular structure of [Yb(Form)I(thf)2]2
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Keywords or phrases (comma separated):

Formamidine, Rare earth, organolanthanoid, Pseudo-Grignard

Welcome Function, Poster Session, Exhibition - Board: 1007 / **43**

Structural characterisation of the retromer complex and associated sorting nexins

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Retromer is a peripheral membrane protein complex that plays a critical role in a broad range of physiological, developmental and pathological processes including Wnt signalling, toxin transport and amyloid production in Alzheimer's disease. The mammalian retromer complex consists of a core heterotrimeric cargo recognition sub-complex (VPS26, VPS29 and VPS35) associated with a dimer of proteins from the SNX–BAR sorting nexin family that drives membrane deformation and tubulation. By recruiting the cargo-selective sub-complex to the forming tubules, the SNX–BAR coat complex mediates the retrograde transport of proteins from endosomes to the trans-Golgi network. Recent studies, however, have highlighted the molecular and functional diversity of retromer and the identification of new interacting proteins has revealed that the role of retromer extends to endosome-to-plasma membrane sorting and regulation of signalling events. Emerging evidence indicates that cargo specificity is mediated by specific sorting nexins. These include SNX3, involved in the trafficking of the Wntless/MIG-14 protein, and SNX27, a PX-FERM protein that mediates the retrieval of the β2-adrenergic receptor.

Using the MX and SAXS/WAXS beamlines at the Australian Synchrotron, we have acquired structural data to determine how the core cargo recognition sub-complex assembles and to characterise the retromer-associated sorting nexins. We are using this information in combination with biochemical and biological studies to understand retromer-mediated endosomal protein sorting and how this fascinating protein complex contributes to a diverse set of cellular processes.

Keywords or phrases (comma separated):

retromer, sorting nexins, SAXS, X-ray crystallography

Welcome Function, Poster Session, Exhibition - Board: 113 / **45**

Composition dependent annealing kinetics of ion tracks in natural apatite

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When high energetic heavy ions penetrate a solid, energy is lost predominantly through inelastic collisions with the target electrons. Such interactions can leave narrow cylindrical trails of damage as the ions traverse through the material, termed 'ion tracks'. In minerals such as apatite, track formation can occur as a result of spontaneous fission from naturally occurring uranium inclusions. The highly energetic fragments damage the matrix material in an analogous manner to heavy ions penetrating a solid, resulting in these so called 'fission tracks'. Upon exposure to elevated temperatures, these tracks are known to reduce in size and the crystalline structure is restored.

In this study, the annealing kinetics of ion tracks (simulating fission tracks) in natural apatites with different F/Cl ratios are investigated using synchrotron based small-angle x-ray scattering (SAXS) combined with ex situ isochronal annealing experiments. Results show that track structure resembles that of cylinders with a constant density and sharp boundaries consistent with amorphous ion tracks. Annealing leads to a reduction in the track radii and the annealing behaviour clearly differs for the four compositions studied. Activation energies for the recrystallization process were extracted from the data obtained.

The results have relevance for fission track geo- and thermochronology.

Keywords or phrases (comma separated):

ion tracks, natural apatite, SAXS

Welcome Function, Poster Session, Exhibition - Board: 305 / **119**

X-ray photoemission spectroscopy of radiosensitizers

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Radiosensitizers are used in radiotherapy to enhance tumour control of radioresistant hypoxic tumours. Recent studies indicate that the formation of radical anions is a key step. Thus understanding the ionization reactions of radiosensitizers is crucial in evaluating the radiosensitization potential and in developing new and more effective drugs. The present study concentrates on the electronic structures of several important radiosensitizers such as nimorazole, 1-methyl-5-nitroimidazole, and 4(5)-nitroimidazole using gas phase synchrotron source X-ray photoemission spectroscopy and quantum mechanics. Detailed analysis of valence and core level spectra will be provided and discussed in the light of possible tautomerism in these compounds.

Keywords or phrases (comma separated):

XPS, radiosensitizers, conformers, experiment and theory

Welcome Function, Poster Session, Exhibition - Board: 405 / **46**

SAXS study of etching behavior of ion tracks in apatite

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Ion tracks consist of narrow (~10 nm), long (~10-100 μm) cylindrical defect regions that are generated by high-velocity heavy ions when they pass through a variety of solids. Such tracks result naturally from fission of uranium inclusions in minerals such as apatite and zircon and are used for determining the age and thermal history of geological material. This so called 'fission track dating' technique utilizes chemical etching to enlarge the tracks to micrometer widths, which enables imaging by optical microscopy and subsequent statistical analysis of their number and length distributions.

The present work investigates how differences in the un-etched track morphology translate into etched ion track dimensions. Tracks were generated by irradiation of the samples with 185 MeV Au ions and 2.3 GeV Bi ions. The morphology of etched and un-etched tracks was studied using synchrotron based small angle x-ray scattering (SAXS) and microscopy techniques such as scanning electron microscopy (SEM) and atomic force microscopy (AFM). Additionally, track annealing was investigated using SAXS in combination with ex-situ annealing experiments performed prior to chemical etching. Results indicate, that the etching process is highly anisotropic, yields faceted etch pits and depends on the mineral composition. These results provide important input to develop an understanding of the correlation of etched and un-etched fission tracks and the use of SAXS as a tool for studying etched tracks.

Keywords or phrases (comma separated):

Ion track, etched, unetched, SAXS

Welcome Function, Poster Session, Exhibition - Board: 110 / **49**

Time-resolved phase evolution during creation of nanoporous Cu current collectors by a dealloying approach

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Dealloying, used to fabricate nanoporous metals, is a process where less noble components (e.g. Al) in the precursor (e.g. AlCu) are dissolved, leaving the nobler elements (e.g. Cu) to form a nanoporous structure. The three-dimensionally nanoporous Cu is desired in lithium-ion batteries as current collectors, which has a unique advantage in providing large surface area for active materials and can accommodate structural strain during lithiation/delithiation reactions. From the perspective of phase evolution, the phases may evolve from Al-Cu phases (fcc &alpha-Al(Cu), tetragonal Al2Cu, monoclinic AlCu or combination of them) to fcc Cu.
In-situ laboratory X-ray Diffraction (XRD) and ex-situ synchrotron XRD experimentation have been implemented to characterise the dealloying of different Al-Cu precursors in preparing fcc Cu. Experiments were carried out using Al75Cu25 (&alpha-Al(Cu) and Al2Cu) and Al65Cu35 (Al2Cu and AlCu) alloys. In the case of in-situ lab XRD experimentation, results showed that for Al75Cu25, the disappearance of &alpha-Al(Cu) and Al2Cu, and the formation of Cu began simultaneously, while for Al65Cu35, the dealloying of Al2Cu and AlCu happened in sequence with the formation of Cu. The highly resoluted ex-situ synchrotron results not only confirmed lab XRD observations, but also

showed transient phases during dealloying for the first time. This study is a model example to investigate the underlying dealloying mechanism from the perspective of phase evolution, and can provide guidance for the development of nanoporous Cu current collectors for batteries.

Keywords or phrases (comma separated):

Dealloying, Nanoporous metals, In-situ phase evolution, Transient phases

Welcome Function, Poster Session, Exhibition - Board: 804 / **154**

Use of an Optical Microscope method to calculate the PVDR's for Synchrotron MRT

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The radiobiology of microbeam radiation therapy (MRT) is poorly understood and this is confounded by difficulties in measuring the dose-distribution. Our investigation assesses the use of microscopy to determine the peak and valley dose in Synchrotron microbeam radiation therapy (MRT). MRT is performed using the horizontal collimator with arrays of 25μm wide x-ray beams with a pitch of 175μm. Three types of Gafchromatic films were irradiated due to their differences in dynamic range. In addition, each type of film was imaged on a series of microscopes. Key parameters for imaging the irradiated film were bit depth and spatial resolution of the microscope camera. Greyscale images taken with and without filters and full colour images were all compared. Also considered was the imaging workflow, with the option for automated loading and imaging of samples an important factor in reducing user error. In conclusion, it was found that a greyscale microscope camera with a bit depth of 14 and pixel size of 0.32μm at a 20x objective was ideal to capture the characteristic of the microbeams.

Keywords or phrases (comma separated):

MRT, microscopy

Welcome Function, Poster Session, Exhibition - Board: 114 / **152**

A combined experimental and computational approach to understanding and developing solid-state ionic conductors

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Materials that exhibit significant mobility of different types of charge carriers have potential applications as fuel-cell membranes, electrodes, batteries and sensors. A thorough understanding of the fundamental atomic-scale mechanisms of the conduction processes in these materials is necessary to identify ways in which their local chemistry and structure can be modified to lower activation barriers and optimize pathways for conduction.
Inelastic neutron scattering experiments will be performed to probe structural fluctuations that may trigger or facilitate the diffusion process. Together with ab initio molecular dynamics (MD) calculations these results will then be used to develop and rigorously verify classical force fields for

empirical calculations to extend the simulations to timescales required to observe actual conduction processes. The results will suggest ways in which the local chemistry and structure of materials can be modified to lower activation barriers and optimize the pathways for ionic conduction.
Good oxide-ionic conductivity, simple chemical composition and the scope for chemical and structural modification make apatite-type Nd9.33Si6O26 a good initial target system. Nd9.33Si6O26 crystallizes in the hexagonal apatite structure P63/m and has 6.7% Nd vacancies located at the 4f site only. A thorough analysis of different arrangements of Nd vacancy positions has been performed to obtain a suitable input model for ab initio MD calculations. Possible arrangements have been classified by the corresponding sum of distances between the vacancies to quantify the degree of distribution and investigate the influence of different vacancy distributions on subsequent calculations.

Welcome Function, Poster Session, Exhibition - Board: 212 / **153**

Australian Synchrotron User Portal

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The User Portal facilitates all aspects of user engagement with the synchrotron from proposal submission, down to processing data on massive and inputting subsequent publications. Ongoing development aims to enhance user experience, improve access to data and increase reporting output; for the users, the Australian Synchrotron and funding bodies.

Keywords or phrases (comma separated):

user, portal, software

Welcome Function, Poster Session, Exhibition - Board: 1013 / **150**

Reconciling Synchrotron SAXS Data with NMR Data for a Two-Domain Protein

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A preliminary structure of the protein Yih1 (Yeast impact homologue 1), a protein of partially characterised function, has been obtained by multi-dimensional NMR methods. The ~300-residue protein has two distinct domains of approximately 120 and 160 residues with an approximately 20-residue linker. However, no NOEs could be found involving contacts between the two domains. Solution-state SAXS data were recorded at the Australian Synchrotron. The structure is clearly monomeric. The NMR structure of one domain was then translated and rotated relative to the other domain until a remarkably good fit to the distinctive SAXS data was obtained. The interaction between the two domains was then, and only then, inspected and found to involve a fairly loose and not implausible association via a small hydrophobic patch and several potential salt bridges. Residual dipolar coupling measurements are in progress to determine alignment vectors of the two domains, and confirm, or otherwise, the accuracy of the SAXS model of domain association.

Cambiaghi TD, Pereira CM, Shanmugam R, Bolech M, Wek RC, Sattlegger E, Castilho BA. 2014. Evolutionarily conserved IMPACT impairs various stress responses that require GCN1 for activating

the eIF2 kinase GCN2. Biochem Biophys Res Commun 443: 592-597 (doi: 10.1016/j.bbrc.2013.12.021).

Keywords or phrases (comma separated):

SAXS, structure modelling, protein NMR structure, solution state

Welcome Function, Poster Session, Exhibition - Board: 211 / **151**

Computing developments and tools supporting beamline science at the Australian Synchrotron

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The Scientific Computing and IT group at the Australian Synchrotron develops software tools to support beamline science, maximise the user experience and accelerate the scientific outcomes of their beam time. Our suite of open source tools facilitate better and more streamlined data collection integrated with automatic and real-time processing, the results of which can inform decisions about further data collection during the user's beam time, optimizing the data they can collect at one visit. In addition, we have developed stand-alone data analysis workflow tools designed for processing and analysis of data, both at the facility and post-experiment at the user's home institute.

Keywords or phrases (comma separated):

Computing

Welcome Function, Poster Session, Exhibition - Board: 1002 / **89**

Binding energy spectra of methoxyphenols: Theory and experiment

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Methoxyphenols (MPs) are antioxidants play an important role in degenerative diseases & cancers. Methoxyphenols are found in many food products however, the molecular details of methoxyphenols are limitedly known. o-methoxyphenol (oMP), m-methoxyphenol (mMP) and p-methoxyphenol (pMP) are positional isomers of one another. The electronic structures, properties and spectra of oMP, mMP & pMP were studied quantum mechanically. The impacts that the methoxy and hydroxy functional groups have on the structure and properties of the MPs were revealed in gas phase. The geometries, ionization energies, ionization spectra & molecular orbitals were investigated and validated against synchrotron sourced experiments completed at the Elettra Sincrotrone. The electronic structure and properties of oMP, mMP and pMP were revealed by systematic studies on the functional groups in gas phase. The results presented indicate that the inner shell of each MP is dominated by the relative functional group positioning and the phenyl aromatic ring buffers the changes induced by the functional groups which stabilizes the MPs in gas phase. It has been established that the valence ionization spectra have conformation dependent changes in the energy range from

12.5eV to 20eV that can differentiate between each isomer. The HOMO-2 electron density distribution changes significantly between each MP. Ionization energy of the MPs all exhibit functional group dependent peaks in C1s and O1s spectra. It will be shown that as the frontier orbitals and outer valence space of the MPs show signature orbital configurations, the frontier orbitals can be used as fingerprint markers for each MP.

Keywords or phrases (comma separated):

DFT, ADF, Methoxyphenol, Binding Energy, Ionization, electronic, structure

Welcome Function, Poster Session, Exhibition - Board: 204 / **88**

The Reference Foil Calibration for the Soft Xray Beamline

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The soft X-ray beam line has fully implemented a series of reference materials that can be placed into the X-ray beam at the same time as a sample is recorded with NEXAFS. This allows a direct cross check against a calibrated photon energy for every spectrum. All the measured reference spectra are presented here in time this will be transparently integrated into the user data taking

Keywords or phrases (comma separated):

Instrumental

Welcome Function, Poster Session, Exhibition - Board: 401 / **19**

Thermal Expansion of Monoclinic Natrojarosite: A Combined Time-of-Flight Neutron and Synchrotron Powder Diffraction Study.

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Jarosites and related minerals are of great interest to a range of mineral processing and research applications. In some settings jarosite formation is encouraged, In other environments jarosite formation can hinder the desired reaction. Jarosites are a major component of acidic soils and are present in significant amounts in acid mine drainage environments. There has been a recent resurgence in interest in jarosite minerals since their detection on Mars. In this context, the presence of jarosite has been recognised as a likely indicator of the presence of water on Mars in the past. It is hoped that study of their formation mechanisms, stability and thermoelastic properties will provide insight into the environmental history of Mars as well as informing terrestrial industrial concerns. To this end we are engaged in a program to study jarosites and their formation and stability behaviour over a range of conditions.
This contribution describes in situ powder diffraction experiments to determine the thermal expansion of a deuterated natrojarosite. Data were collected on the HRPD beamline at the ISIS spallation source where the natrojarosite sample was heated from 10–700K, and at the powder diffraction beamline at the Australian synchrotron where the sample was heated from 80-700K.

Thermal expansion coefficients have been fitted from 10-470K and show that there is most variation normal to the layers of sulphate tetrahedra and iron octahedra within the jarosite structure and contains more flexible hydrogen bond linkages. Details of the combined neutron-synchrotron data analysis approach will be discussed.

Keywords or phrases (comma separated):

Earth Sciences, Jarosite, Mars, Synchrotron, Neutron, Diffraction, in situ

Welcome Function, Poster Session, Exhibition - Board: 301 / **97**

DNA Binding Studies using Synchrotron Radiation Circular Dichroism (SRCD) data and Mathematica

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The binding affinity of a series of square planar achiral platinum(II) compounds of the type [Pt(AL)(IL)]2+, where AL is 1,2-diaminoethane and IL are 1,10-phenanthroline (phen), 4-methyl-1,10-phenanthroline (4-Mephen), 5-methyl-1,10-phenanthroline (5-Mephen), 4,7-dimethyl-1,10-phenanthroline (47-Me2phen), 5,6-dimethyl-1,10-phenanthroline (56-Me2phen) or 3,4,7,8-tetramethyl-1,10-phenanthroline (3478-Me4phen) has been reinvestigated using Synchrotron Radiation Circular Dichroism (SRCD) spectroscopy. SRCD spectroscopy uses the intense light of a synchrotron beam to measure shorter wavelength data, exposing CD bands not previously observed. The additional peaks were considerably more intense than those observed by conventional CD spectroscopy, and could be used for binding affinity determinations. In addition, the authors have reviewed the various mathematical approaches used to estimate equilibrium binding constants and thereby demonstrate that their mathematical approach, which has been implemented with Wolfram Mathematica, has merit over other methods.

Keywords or phrases (comma separated):

Synchrotron Radiation Circular Dichroism (SRCD), platinum(II)

Welcome Function, Poster Session, Exhibition - Board: 502 / **94**

Unraveling the morphology of a novel, high-efficient polymer solar cell using synchrotron-based techniques

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Organic solar cells are a next generation photovoltaic technology with the potential for a low cost of manufacturing and printing on flexible substrates. The efficiency of organic solar cells has increased rapidly, recently exceeding 9% efficiency. Understanding the morphology of the active layer of polymer based bulk heterojunction solar cells is necessary to further improve device performance. Here we comprehensively study the morphology of a novel electron donor polymer PBDTTT-EFT with efficiency of over 9%. The orientation and microstructure of the neat polymer films and in blend films with PC71BM are examined by using a combination of surface-sensitive near edge X-ray absorption fine structure (NEXAFS) spectroscopy from the soft X-ray beamline and bulk sensitive

grazing incidence wide angle X-ray scattering (GIWAXS) collected at the SAXS/WAXS beamline. In the blend, a "face-on" orientation of PBDTTT-EFT is observed with π-π stacking normal to the substrate in the bulk of thin film, while a more "edge-on" orientation with side-chain-stacking normal to substrate is observed at the surface of the film. In organic solar cells, face-on structures enhance charge transport in the critical vertical direction. An edge-on orientation of PBDTTT at the hole extracting interface is not considered to be ideal for charge collection, but does not appear to adversely affect device performance. The additive 1,8-Diiodooctane is also used to improve the crystallization of PBDTTT-EFT and to control the aggregation of PC71BM.

Keywords or phrases (comma separated):

Organic solar cells, morphology, GIWAXS, NEXAFS,

Welcome Function, Poster Session, Exhibition - Board: 1006 / **93**

Structure-based Development of Inhibitors of HCV NS5b

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Infection by the Hepatitis C virus (HCV) affects in the order of 150 million people world-wide with more than 300,000 dying each year from HCV-induced liver disease. The RNA-dependent RNA-polymerase of HCV, NS5b, is widely accepted as an ideal candidate for therapeutic development due to the lack of an equivalent enzymatic activity in normal human cells and the absolute dependence of viral replication on NS5b. Here we present the results of a fragment-based discovery program carried out as part of our research into NS5b inhibitors. A number of fragments identified as NS5b ligands through STD-NMR analysis, as well as structural analogues of these fragments, were soaked into crystals of HCV NS5b and the structures of the complexes determined. On the basis of the proximity of one of these compounds to the primer grip site of the enzyme, hybrid compounds linking the compound and a known inhibitor were designed and synthesised. These hybrid compounds were found to be potent inhibitors of the HCV replicon and NS5b enzymatic assay and were shown crystallographically to bind in the primer grip site in precisely the orientation predicted from modelling.

Keywords or phrases (comma separated):

Structure-based drug design, Fragment screening, Antiviral

Welcome Function, Poster Session, Exhibition - Board: 303 / **90**

Fast Fluorescence Tomography: Challenges and Opportunities

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The promise of non-destructive 3D elemental imaging using x-ray fluorescence tomography is alluring, but the technique is not widely utilised due to the extremely long scan times required for

modest tasks. Accordingly, the technique is often applied to imaging of small specimens at low definition. Our first 3D demonstration on a 10-m estuarine diatom, Cyclotella meneghiniana achieved a resolution of around 400 nm over 1003 voxels (1 Mvox) with 24 projections. More recent developments at the Australian Synchrotron have used a KB mirror pair and the Maia detector system to achieve extremely high pixel rates. In addition to its extremely high speed, the Maia detector is very efficient, employing a novel back-scatter geometry to achieve a very large 1.2-sr solid angle. Using the Maia detector system, measurements can properly sample the sinogram and have achieved 175 Mvox with scan times of around 14 hours.

Detailed tomographic measurements of large (mm-scale) and whole organisms are now routine. Reductions in measurement time and radiation exposure further enable imaging of a whole class of specimens that are susceptible to radiation damage and/or intolerant of measurement conditions. We have recently demonstrated fast cryo tomography and XANES tomography on biological specimens, with tantalising results. We present several applications and describe several challenges that face this growing field.

Keywords or phrases (comma separated):

X-ray fluorescence microscopy, XANES, tomography, self absorption

Welcome Function, Poster Session, Exhibition - Board: 112 / **98**

Mechanistic Studies of Catalytically Relevant On-Metal N-Heterocyclic Carbene Transformations

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N-Heterocyclic Carbenes (NHCs) have many advantages to their phosphine analogues and have been used for a variety of catalytic applications. Bis-NHCs in particular have been used where bidentate phosphine complexes have previously been used including polymerisation and cross coupling reactions. Our group has been interested in palladium bis-NHCs with a wide variety of different N-substituents and linker lengths for catalytic applications.

We recently discovered an unexpected ligand rearrangement for a bis-NHC palladium complex when reacting the ethylene linked N-mesityl imidazolium salt with Pd(OAc)2. Along with the expected chelate formation an unusual C-C coupling product was also formed. DFT calculations indicate the formation is through the decomposition of a normal - abnormal intermediate via an on-metal ligand rearrangement. Given the extensive usage of this ligand class in catalysis and the decomposition route leading to this product it is important to understand and thus prevent its formation.

DFT investigations established will be presented as well as synthetic studies probing the mechanism. Symmetrical analogues have been synthesised as well as unsymmetrical analogues blocking the C2 position.

An alternative mechanism has also been explored for the decomposition of the chelate product to the C-C coupled product via the formation of a 1:2 ligand: palladium species. The NHC bridged dipalladium product has been isolated and structurally confirmed by X-ray crystallography and shown to offer high yielding access to C-C coupled decomposition products.

Keywords or phrases (comma separated):

organometallics, catalysis, crystallography, palladium

Welcome Function, Poster Session, Exhibition - Board: 306 / **99**

Using synchrotron radiation to determine the X-ray structure and ct-DNA binding affinity of platinum(II) anticancer complexes

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Platinum(II) anticancer complexes incorporating 2,2'-bipyridine (bpy), 4,4'-dimethyl-2,2'-bipyridine (44Me2bpy) and 2-(2'-pyridyl)quinoxaline (2pq) as polyaromatic ligands and cyclic diamines as ancillary ligands have been synthesised and were characterised via several methods including synchrotron radiation X-ray crystallography. The crystal structure of [Pt(44Me2bpy)(1S,2S-diaminocyclohexane)]2+ (44MEBSS) revealed a square planar coordination geometry similar to other complexes of this type whereas the complex [Pt(2pq)(1S,2S-diaminocyclohexane)]2+ (2PQSS) was distorted square planar.. The binding of 2PQSS and 44MEBSS to calf-thymus DNA (ct-DNA) was analysed using synchrotron radiation circular dichroism (SRCD) melting experiments and compared to similar complexes that incorporate 1,10-phenanthroline and dipyrido[3,2-f:2',3'-h]quinoxaline. The results revealed unexpected trends in DNA affinity relative to polyaromatic ligand size.

Keywords or phrases (comma separated):

synchrotron radiation circular dichroism (SRCD) melting experiments

Welcome Function, Poster Session, Exhibition - Board: 603 / **67**

3D Strain Characterisation in Nanodiamonds using Bragg Coherent Diffractive Imaging (BCDI)

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Nanodiamonds with nitrogen vacancy (N-V) centres have been shown to be useful for applications involving cellular tracking in vivo at the molecular level[1]. The sustained fluorescence of these nanodiamonds is related to their structure, and is supposed to be influenced by the strain distribution inside the crystals. In the present work, Bragg coherent diffractive imaging (BCDI) has been employed for three-dimensional (3D) strain mapping of single-crystal synthetic nanodiamonds dispersed onto a silicon substrate. Defects were introduced in these isolated nanodiamonds by implanting with 2 MeV protons to a dose of of 1015 H+ ions.cm-2 using a palletron accelerator1. The resulting strains were mapped using BCDI in order to characterise the 3D deformation field within the individual NDs.

[1] C.-C. Fu, H.-Y.Lee, K. Chen, T.-S.Lim, H.-Y.Wu, P.-K.Lin, P.-K.Wei, P.-H.Tsao, H.-C. Chang, W. Fann, Proceedings of the National Academy of Sciences, 104(3), p. 727-732, (2007)

Keywords or phrases (comma separated):

3D Strain Mapping, Nanodiamonds, Bragg Coherent Diffraction Imaging (BCDI)

Welcome Function, Poster Session, Exhibition - Board: 1102 / **66**

Near-edge X-ray absorption fine-structure spectroscopy of naphthalene diimide-thiophene co-polymers

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Near-edge X-ray absorption fine-structure (NEXAFS) spectroscopy is an important tool for probing the structure of conjugated polymer films used in organic electronic devices. High-performance conjugated polymers are often donor-acceptor co-polymers which feature a repeat unit with multiple functional groups. To facilitate better application of NEXAFS spectroscopy to the study of such materials, improved understanding of the observed NEXAFS spectral features is required. In order to examine how the NEXAFS spectrum of a donor-acceptor co-polymer relates to the properties of the sub-units, a series of naphthalene diimide-thiophene-based co-polymers have been studied where the nature and length of the donor co-monomer has been systematically varied. The spectra of these materials are compared with that of a thiophene homopolymer and naphthalene diimide monomer enabling peak assignment and the influence of inter-unit electronic coupling to be assessed. We find that while it is possible to attribute peaks within the pi* manifold as arising primarily due to the naphthalene diimide or thiophene sub-units, very similar dichroism of these peaks is observed indicating that it may not be possible to separately probe the molecular orientation of the separate sub-units with carbon K-edge NEXAFS spectroscopy

Keywords or phrases (comma separated):

NEXAFS, conjugated polymers, solar cells

Welcome Function, Poster Session, Exhibition - Board: 501 / **65**

In-situ examination of electrodeposited manganese dioxide electrodes for energy storage applications: a combined small angle x-ray scattering and powder diffraction study.

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Manganese dioxide is used in many electrochemical applications including catalysis, batteries and electrochemical capacitors. Thin film manganese dioxide electrodes prepared by electrodeposition have been shown to have extremely high capacitive performance [1]. This is due to manganese dioxide thin films having low resistance and high surface area. However, these properties diminish markedly as film thickness increases. Hence, understanding how the deposition mechanism causes this effect is vital for improving material performance.
In order to determine the relationship between deposition mechanism, material properties and electrochemical performance, thin film formation under different deposition conditions must be examined. In this work, the deposition of manganese dioxide was examined in-situ using two different methods; namely small angle X-ray scattering (SAXS) and powder diffraction (PD).
The morphology of manganese dioxide thin films is expected to affect their performance as it influences porosity and surface area. SAXS was used to characterise the surface morphology of manganese dioxide, specifically features such as pore size and particle size in the < 1 nm size range.

The crystal structure also affects the electrochemical performance of manganese dioxide via the conductivity (ionic and electronic) of different crystal phases of manganese dioxide [2] . The crystal structure of manganese dioxide films was also examined as a function of the deposition time and electrolyte composition. The crystal structure and its effect on capacitive performance is also investigated.
[1] A. Cross, et al., J. Power Sources, 196 (2011) 7847-7853
[2] M.F. Dupont, et al., J. Electrochem. Soc. 160 (2013) A1219-A1231

Keywords or phrases (comma separated):

electrochemical capacitors, manganese dioxide, thin films, electrodeposition , SAXS, powder diffraction

Welcome Function, Poster Session, Exhibition - Board: 203 / **63**

Calorimetry for Synchrotron radiation

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Calorimetry is the most accurate method of quantifying the x-ray flux and dose in the synchrotron beam lines for medical and research applications. In calorimetry, the radiation dose absorbed results in raising the temperature of the absorbing medium which is measured accurately. Several absorbing media have been tried but graphite with relatively low specific heat and zero heat defect has been the chosen medium for calorimetry.
Graphite calorimeters are dose-rate independent and hence can be used over wide dynamic range of intense synchrotron x-ray beams. They are energy-independent and hence are ideally suited to measure the total energy fluence over the entire low energy photon spectrum compared to diodes, films etc. The temperature rise due to the absorbed dose is high (of the order of several milli-kelvins) resulting in simple read-out instrumentation.
The design criteria of the ARPANSA graphite calorimeter which was successfully used to measure the high dose-rates (~2500 Gy/sec) in Hutch1B and (~50 Gy/sec) in Hutch 3B at the Australian Synchrotron is presented in this talk. The need for absorbed dose to water is realised through Monte Carlo calculations of the conversion factor from the graphite absorbed dose measured by the calorimeter. The talk also includes the different calorimeters used earlier internationally for photon fluence measurements different from the unique application of the ARPANSA calorimeter for the IMBL measurements.

Keywords or phrases (comma separated):

calorimetry IMBL absolute dosimetry

Welcome Function, Poster Session, Exhibition - Board: 608 / **62**

Wrestling with Big Data at IMBL

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It is now possible to collect very rapid micro-CT scans at IMBL with data collection times down to 16s or less per scan. This makes it possible to collect three-dimensional data on physical systems that are changing over time.

While this provides an exciting scientific opportunity it also comes with significant challenges in terms of data storage, reconstruction and analysis. Dealing effectively with such 'Big data' challenges is increasingly important in order to ensure that we derive the most benefit from the opportunity that rapid data collection offers.

The authors recently acquired a large number of time-resolved datasets following the development of bread dough during proving and baking. The aim of the experiment was to determine differences in the rheological and machanical properties of dough made using different flours and salt additives.

This experiment resulted in over 460 datasets which required reconstruction and analysis and provided a good test-case for addressing the big-data challenge. This challenge has been addressed using tools available (and new tools developed) on the MASSIVE supercomputer cluster.

We outline the challenges and how they were addressed as well as providing some reflection on what might be needed in the future to meet our growing data needs.

Keywords or phrases (comma separated):

Big data, IMBL, micro-CT, time-resolved, bread

Welcome Function, Poster Session, Exhibition - Board: 1003 / **60**

Developing Bragg Coherent Diffractive Imaging for Biological crystals at the Advanced Photon Source

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Bragg Coherent Diffractive Imaging (BCDI) of radiation hard crystals (e.g. lead, gold) is now a relatively mature technique for characterising elastic strain fields at the nanoscale. The technique relies upon the fact that coherent diffraction from a crystal produces a continuous intensity distribution around every Bragg peak; if sampled correctly, this information can be used to reconstruct the crystals three-dimensional complex density. The corresponding phase of the density is related to the elastic strain which provides information on the atomic displacements within the crystal lattice (Pfeifer et al, 2006).
For protein crystals the presence of strain and disorder within the crystal can have a critical influence on the size of crystal that can be grown and the quality of diffraction data that can be collected. Hence, our group has recently been applying BCDI to the high-resolution characterisation of elastic strains in micron-sized protein crystals. One of the major experimental challenges with such samples is that they are highly radiation sensitive; however the combination of cryogenic temperatures and photon counting detectors offers a possible solution.
Here we present the first BCDI results collected from protein crystals of Lysozyme measuring ~3.5 microns in diameter using the newly developed cryo-BCDI setup at beamline 34-ID-C at the Advanced Photon Source. Our preliminary reconstructions from the BCDI data are promising, and pave the way for strain analysis of crystals composed of more complex proteins in future experiments.
Pfeifer, M. et al(2006).Nature,442(7098),63–6.

Keywords or phrases (comma separated):

Strain mapping, coherent imaging

Welcome Function, Poster Session, Exhibition - Board: 901 / **36**

LCP crystallisation and its challenge with the D2L receptor

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Self-assembly lipids have been used to solve a number of G-protein coupled receptor structures to date and the mechanism behind it still remains a mystery. Here we report on two factors; lipid incubation time and protein concentration and investigate three different lipid systems; monoolein (MO), phytantriol (PT) and phytanoyl ethanomide (PE), which influenced the uptake of the Dopamine 2 long (D2L) receptor into the lipid structure. We show reasons why the D2L receptor has proven to be difficult to crystallize in the commercial cubic phase lipid, MO, and show why we need to develop novel crystallization media.

Keywords or phrases (comma separated):

GPCR, LCP crystallization, lipids, D2 receptor

Welcome Function, Poster Session, Exhibition - Board: 1101 / **31**

Air-Stable Electron Depletion of Bi2Se3 Using Molybdenum Trioxide into the Topological Regime

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Topological insulators, such as Bi2Se3, are a new class of material that possess topologically protected Dirac surface states that hold great promise for next generation nano-electronic devices [1]. However, major challenges exist in realizing Bi2Se3 devices that operate in the topological regime in air. The first is that as-prepared Bi2Se3 is invariably n-type doped due to selenium vacancies [1], where the Fermi level resides in the bulk conduction band, not within the Dirac surface states necessary to realize these electronic devices. The second is that Bi2Se3 when exposed to atmosphere becomes further n-type doped and degrades over time [2].

Utilizing high-resolution photoelectron spectroscopy on in-situ cleaved Bi2Se3 single crystals we demonstrate that the strong electron acceptor molybdenum trioxide (MoO3) is capable of depleting ~1013 cm-2 electrons from Bi2Se3 to place the Fermi level well within the topological regime. We implement a doping model based on Fermi-Dirac statistics to accurately describe the doping behaviour as a function of MoO3 coverage. Furthermore, in-situ transport measurements on MBE grown Bi2Se3 films are used to demonstrate that a 100 nm film of MoO3 is also capable of protecting Bi2Se3 from degradation upon exposure to atmosphere and further n-type doping [3].

References:
[1] Y. Xia, et al., Nature Physics 5, 398 (2009)
[2] D. Kong, et al., ACS Nano 5, 4698 (2011).
[3] M. T. Edmonds, et al., ACS Nano 8, 6400 (2014).

Keywords or phrases (comma separated):

Bi2Se3, Topological Insulator, Molecular Doping

Welcome Function, Poster Session, Exhibition - Board: 104 / **32**

Structural properties of Indium and Indium + Carbon implanted Germanium

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Ge has been considered as a potential alternative material for silicon in fabricating future advanced CMOS devices due to its high hole mobility and low dopant activation temperature. Here we study the effect of In concentration on the structural and electrical properties of Ge with or without C co-implantation. By using extended x-ray absorption fine structure and x-ray absorption near-edge spectroscopy, we found that in the case of In implanted Ge, In atoms occupy a substitutional site in the Ge lattice with In concentration ≤ 0.3 at. %, yet when In concentration is ≥ 1 at. %, In precipitates to from metallic particles as confirmed by transmission electron microscopy, evidence of an In – Vacancy complex is also apparent with EXAFS. With C co-implantation, x-ray absorption spectra show that In precipitation was suppressed when the In and C concentration are ≥ 1 at. % (also supported by transmission electron microscopy), and evidence of In – C pairing formation was found in EXAFS. Hall Effect measurement also showed that the carrier density significantly increased and In atom activation was improved with C co-implantation. Density Functional Theory was applied to calculate the binding energies of In – In, In – Vacancy and In – C clusters, and it was found that In atoms should prefer to pair with vacancies and C in Ge. Density Functional Theory was also used to simulate the lattice structure of the samples to compare and support the simulated structure from x-ray absorption spectra.

Keywords or phrases (comma separated):

Indium Carbon coimplantation and clustering, Germanium, EXAFS, XANES, TEM, DFT

Welcome Function, Poster Session, Exhibition - Board: 102 / **33**

Ion irradiation induced porosity in Germanium

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Ion irradiation of crystalline germanium (c-Ge) results in the formation of a porous surface, and sometimes buried porous layer. The controlled fabrication of such porous structures has potential applications in lighting, gas detection and catalytic applications. In the present work, we employ a combination of complimentary characterisation techniques to better understand the crystalline-to-porous transformation in Ge as a function of implantation conditions (fluence and temperature). The project is also being extended to include the Si1-x Gex alloy.
Post-implantation optical profiling indicated that significant swelling of the irradiated layer occurs (up to 400 nm), consistent with the formation of a porous surface layer. The optical result also showed evidence of a four stage swelling process with ion fluence for pure Ge, whereas the alloys with different fraction of Ge (90, 80, and 77%) show two different stages of swelling (up to 100 nm). It has been observed by utilizing Raman spectroscopy that the structural properties of the porous layers were also dependent on the ion fluence and temperature as evidenced by changes in the position and width of the characteristic phonon bands. Scanning electron microscopy is also applied to study the morphology as a function of ion fluence and temperature for different concentration of Ge. The pore shape and depth damage distribution has been investigated by using Transition electron microscopy. Small angle x-ray scattering measurements provided further evidence of an implant temperature-dependent.

Keywords or phrases (comma separated):

porosity, ion irradiation, SAXS,SEM,TEM

Welcome Function, Poster Session, Exhibition - Board: 106 / **39**

In situ high-energy x-ray diffraction study on bulk bismuth ferrite ceramic

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Bismuth ferrite, BiFeO3 (BF) is a multiferroic ceramic familiar for the existence of both strong ferroelectric and magnetic ordering at room temperature. In addition to the multiferroicity, the remarkably high Curie temperature (Tc) and spontaneous polarization (Ps) of BF has made it an attractive candidate to replace lead-based Pb(Zr,Ti)O3 for industrial applications. However, the high coercive field (> 10 kV.mm-1) and related poling inefficiency is the major issue for the application of BF as a piezoelectric material. To tailor the actuator property of bulk BF ceramic, it is essential to understand the structure-property relationship in the ceramic at field-on condition. In situ high-energy x-ray diffraction measurements with a large area detector in transmission geometry can be utilized to quantify the structural variation with field in these materials. Here, we used the high-energy x-ray diffraction beamline I12-JEEP of the Diamond Light Source, UK. Qualitative and quantitative analysis of diffraction patterns reveal that ferroelastic domain switching is the primary mechanism for the strain response. Interestingly, a strain magnitude similar to thin film BF was observed; however, the origin of strain response is different.

Welcome Function, Poster Session, Exhibition - Board: 304 / **105**

A combined X-ray and neutron scattering study examining triglyceride digestion

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In the body, triglyceride oils are digested into amphiphilic fatty acid and monoglyceride. These products subsequently self-assemble into a range of structures, including liquid crystal phases.1,2 It is hoped that understanding their digestion and self-assembly processes offers new opportunities for lipophilic drug delivery. Previous studies using small angle scattering and cryo-TEM have identified some of the phases formed during triglyceride digestion.3 Precise details of the digestion process remained an area of uncertainty which the current work aimed to resolve.
Triglyceride digestion has been examined using synchrotron small angle X-ray and neutron scattering as well as chemical methods. Synchrotron SAXS was used for the time-resolved in-situ study of phase formation and phase transitions during digestion. Neutron scattering, on the other hand, allowed the use of contrast variation to identify the specific digestion products involved in different aspects of the phase formation.
Details of the digestion process and the location of the different digestion products within the self-assembled structures will be presented. These results improve our understanding of lipid digestion and provide an excellent case study in the complimentary use of X-ray and neutron methods in the analysis of a single, complex system.

1. Salentinig S., Sagalowicz L., Leser M.E., Tedeschi C., Glatter O. Soft Matter, 2011, 7, 650-661.
2. Salentinig S., Sagalowicz L., Glatter O. Langmuir, 2010, 26, 11670–11679
3. Salentinig S., Phan S., Khan J., Hawley A., Boyd B. ACS Nano, 2013, 23, 10904-10911.

Welcome Function, Poster Session, Exhibition - Board: 406 / **107**

Infrared spectra of H2O aerosols diluted with D2O

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Water is both an important and interesting molecular system that needs to be well understood because it exists in so many fields of research, and more often than not its presence can be undesired. The ice particles in both our own atmosphere, and the ISM, predominantly exist as either small ice particles or silicate dust grains coated with ice which can act as a reaction medium for producing more complex molecules.
We explore how isotopic dilution, using D2O, affects the shape and frequency of the vibrations that are observed in the mid infrared (MIR) spectrum[1], more specifically the OH- and OD- stretching modes. In addition to changing the concentration of D2O used, the physical aspects of the experiment itself also contribute to what is observed. Presented herein, is the MIR data collected at the Australian Synchrotron, using different H2O:D2O ratios, and at temperatures ranging from 6 K to 210 K.

1Medcraft, C, McNaughton, D, Thompson, CD, Appadoo, DRT, Bauerecker, S, Robertson, EG, Phys. Chem. Chem. Phys. 2013, 15, 3630

Keywords or phrases (comma separated):

ice, infrared, aerosol, synchrotron

Welcome Function, Poster Session, Exhibition - Board: 701 / **106**

Service Crystallography through the Industry Group

Alan Riboldi-Tunnicliffe[1]

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We offer services from cloning your gene and small scale expression trials, protein purification and crystallisation trials, through to full structure determination.

Welcome Function, Poster Session, Exhibition - Board: 302 / **101**

Using Synchrotron Radiation Circular Dichroism (SRCD) to probe G-quadruplex DNA-platinum(II) complex interactions

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Platinum(II) anticancer drugs, such as cisplatin and carboplatin, bind to DNA coordinately and have many limitations including poor effectiveness against many cancer cell lines, acquired resistance, cross-resistance as well as unwanted side effects. To overcome these limitations we have recently synthesised dinuclear (2,2':6',2"-terpyridine)-based complexes that are connected by thiol chains of varying length (with IC50 in L1210 cells). These compounds have demonstrated potent cytotoxicity in cancerous cell lines and are thought to interact with DNA through π-stacking interactions involving their terpyridine moieties. Small molecules that selectively bind to G-quadruplex DNA (Q-DNA) have been shown to stabilise these structures, and so Q-DNA represents a potential biological target for the suppression of telomerase activity. Here we present SRCD-based melting studies of the binding of our platinum(II) complexes to Q-DNA.

Keywords or phrases (comma separated):

Synchrotron Radiation Circular Dichroism (SRCD) melting studiesof G-Quadraplex DNA

Welcome Function, Poster Session, Exhibition - Board: 606 / **100**

Improved Dynamic Analysis Method for Quantitative High Definition XFM Element Imaging using Maia

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Projection of quantitative element images from fluorescence data collected on the XFM beamline of the Australian Synchrotron equipped with a Maia detector and real-time processor uses the Dynamic Analysis (DA) method in the GeoPIXE software. It uses matrix transformations to achieve least-squares fitting of pixel spectra in X-ray fluorescence imaging and tomography at up to ~3 x 10^7 events per second in the FPGA processor for real-time imaging or ~2-8 x 10^6 on a desktop, which typically corresponds to about 10^4-10^5 pixels per second. At least initially, it assumes uniform sample composition, background shape and constant model X-ray relative intensities. Our present approach is to then apply an iterative matrix (composition) correction. But this does not account for changing background shape and X-ray relative intensities evolving spatially with significant changes in composition.

A new method, applied in a second pass, uses an end-member phase decomposition obtained from the first pass, and DA matrices determined for each end-member, to re-process the event data with each pixel treated as an admixture of end-member terms. This approach better tracks spatially complex samples as encountered in geological and environmental research while still benefiting from the speed of DA. The decomposition and DA matrices can be applied to a series of samples with similar content. This paper describes the method and illustrates how the enhanced accuracy of spectral deconvolution improves imaging of challenging materials.

Keywords or phrases (comma separated):

XFM imaging, Maia detector, Fluorescence, SXRF, quantitative analysis

Welcome Function, Poster Session, Exhibition - Board: 1008 / **103**

Strain specificity in a fully conserved epitope of a malaria vaccine candidate

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Merozoite surface protein 2 (MSP2) is an unstructured protein of the Plasmodium falciparum merozoite. The two allelic forms of MSP2, 3D7 and FC27, differ in a central variable region which is flanked by conserved N- and C-terminal regions. Vaccine trials using 3D7-MSP2 have shown evidence of strain specific protection despite the detectable presence of conserved region antibodies. This work focuses on an N-terminal epitope recognised by the mouse monoclonal antibody, 6D8. Despite recognising a fully conserved epitope, 6D8 shows strain specificity. Understanding the determinants of 6D8 specificity will assist the designing of a broad-spectrum MSP2-based malaria vaccine. 6D8 was re-engineered into antibody fragments (scFv and Fv) and validated by SPR and ITC. Additionally, a series of N-terminal peptides were synthesised to locate the minimal binding region (NAYNMSIRR, KD = 6 nM) and investigate the strain specificity of 6D8. High-resolution (1.2 Å) crystal structures of four N-terminal peptides bound to 6D8 Fv revealed identical binding mechanisms irrespective of N- or C-terminal extensions from the minimal epitope. However, binding data indicates that the strain specificity of 6D8 to 3D7 and FC27-MSP2 requires the first 5 C-terminal residues of the variable region, and suggest that entropic effects of unbound variable residues determine the strain specificity of 6D8. This progress will underpin the design of effective strain-transcending MSP2-based malaria vaccines and may have wider implications for our understanding of the immune response towards unstructured proteins.

Keywords or phrases (comma separated):

Immune system, Untructured antigens, Malaria, Vaccine, MSP2, Strain specificity

Welcome Function, Poster Session, Exhibition - Board: 803 / **102**

Comparison of dose deposition patterns of multi-slit versus single-slit collimators in synchrotron MRT and their effect on 9L cells

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Results of quantitative evaluation and comparison of dose deposition patterns and radiobiological effects in synchrotron microbeam radiotherapy (MRT) - in particular, the inter-microbeam “valley” dose - for six beam geometries and three different in-beam dose values are presented. The X-Tream dosimeter and Gafchromic film have been used to quantitatively compare the dose distribution resulting from step-and-shoot single versus multi-slit collimation for two of the six beam geometries in order to validate the use of the single-slit approach for the other four geometries. Radiobiological effects have been assessed by evaluating post-irradiation cell survival fractions on cultured 9L gliosarcoma cells.

Preliminary results from our allocated beamtime at the Australian synchrotron show that valley doses obtained with single-slit step-and-shoot collimation are between 3 and 10% lower than in multi-slit collimation with the same beam width and peak-to-peak distance. The biological effects of any difference in spatial and temporal dose deposition patterns were also investigated by irradiating in vitro 9L gliosarcoma cells (at a depth of 2 cm in water, surrounded by solid water) using both beam delivery methods. At the time of writing this abstract, the irradiated cells are in incubation and their colony formation abilities will be assessed in two weeks.

Keywords or phrases (comma separated):

MRT, Step and Shoot collimation, Radiobiological effects, clonogenic assay, dosimetry

Welcome Function, Poster Session, Exhibition - Board: 702 / **109**

Materials Diffraction and Scattering with Industry

Tamsyn Ross[1]

[1] *Australian Synchrotron*

An increasing number of industrial users are looking to the SAXS/WAXS and PD beamlines for their materials characterisation needs. In recent years commercially-relevant projects from a broad range of fields, including primary industry, energy materials and mining, have met with success due to staff expertise and dedication and the high quality of the facilities.

Keywords or phrases (comma separated):

Industry, materials, diffraction, SAXS, WAXS, PD

Welcome Function, Poster Session, Exhibition - Board: 205 / **108**

The Technologies That Drive the MX Beamlines.

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Co-author(s): Alan Riboldi-Tunnicliffe [1] ; Daniel Eriksson [1] ; David Aragao [1] ; Jason Price [1] ; Mark Clift [1] ; Nathan Cowieson [1] ; Nathan Mudie [1] ; Rachel Williamson [1] ; Santosh Panjikar [1] ; Tom Caradoc-Davies [2]

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The MX1 and MX2 beamlines at the Australian Synchrotron are sophisticated machines for the collection of single crystal X-ray diffraction data. The two stations serve a diverse community of researchers in the structural sciences, from mineralogy to virology.
Here we look 'under the hood' at the technologies that bind the individual beamline components together into a highly automated data collection system.
In the last few years a full re-write of several key software components has delivered new code and capabilities for beam attenuation, data collection and energy change and also beamline setup, diffraction rastering and autoprocessing resulting in a more reliable and easier beamline to support.
We are now starting to work on the software that runs our robots to apply a similar improvement.
These upgrades together with the hardware changes will keep both beamlines useful for the most complex biological and chemical questions our researchers need to address

Keywords or phrases (comma separated):

MX1, MX2, Crystallography, Automation, Beamline,

Welcome Function, Poster Session, Exhibition - Board: 903 / 7

Changes to the Nanostructure of Collagen in Skin During Leather Processing

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Leather is a complex biomaterial largely composed of collagen fibrils. As skins are processed to produce leather, chemical and physical changes take place that affect the physical properties of the material. The structural foundation of these changes at the collagen fibril level is not fully understood and formed the basis of this investigation. Synchrotron-based small-angle X-ray scattering was used to quantify fibril orientation and D-spacing through eight stages of processing from fresh green ovine skins to staked dry crust leather. Both these structural aspects were found to change with processing. At a higher pH, both D-spacing and the fibril orientation index are lower. The elastic modulus also changes with high salt concentrations and low pH conditions associated with materials that have a low elastic modulus. This study shows that there are structural changes taking place during the processing of skin to leather. It is proposed the change in D-spacing is due to pH affecting the H-bonding within the tropocollagen unit and the decrease in OI is due to the relaxation of tension in the fibrils enabling the collagen fibrils to bend or distort more. This understanding informs the influence of the chemistry at different stages of processing on the development of the final physical characteristics of leather. By understanding the structural changes of collagen that occur when skin is subjected to chemical and mechanical treatments, it may be possible to modify some of these processing steps to alter the final properties of leather.

Keywords or phrases (comma separated):

collagen, SAXS, leather, structure

Welcome Function, Poster Session, Exhibition - Board: 201 / **84**

A Model-based approach to Motion Control design at the Australian Synchrotron

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Reliable, robust, and predictable control of motion axes is a key component to any synchrotron beamline. To this end, new motion controls hardware (Geo-Brick-LV-GBLV, Delta-Tau-UK) was recently introduced at the XAS, IMBL, SXR and XFM Beamlines. Challenges included optimising and tuning motion axes behaviour for in-vacuum motors, closed-loop tracking axes, and scanners with velocity requirements.
Generally, the GBLVs are very powerful motion controllers, which can be utilised for many different applications by soft configuration/tuning. This sophistication and flexibility often implies complex and potentially difficult configuration and tuning processes by highly specialized engineers, particularly for non-trivial motion environments at beamlines.
In order to address these challenges, a model-based approach was used to provide a framework for generalising and formulating the motion control system. The model makes it possible to classify applications (e.g. positioning, scanning), and suggest optimum motor configuration and tuning based on design inputs of motor and stage specification and user requirements (e.g. accuracy, speed).
The model developed has been applied successfully to more than 50 motion axes at XAS, IMBL and SXR, adequately predicting motor performance outcomes. It strongly improves the classical approach of system design based on expected configurations and manual tweaking in the field. This is because a model-based solution documents and incorporates learning, evolves to handle more and more complex systems, classifies problem types, and, importantly, reduces non-standardisation while improving usability, which ultimately benefits the user community through more effective beamline operation.

Keywords or phrases (comma separated):

Motion Control, GeoBrick, PMAC, Model-Based design, Modelling

Welcome Function, Poster Session, Exhibition - Board: 1004 / **81**

Beyond SAXS, how do we predict misfolding hotspots in alpha-synuclein?

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The 140 residue intrinsically disordered protein -synuclein (-syn) misfolds to form fibrils that are the major constituent of the Lewy body intracellular protein inclusions and neurotoxic oligomers occurring in a number of neurodegenerative diseases, including Parkinson's disease (PD) and dementia

with Lewy bodies. Using SAXS data analysed by ensemble optimised modelling (EOM) we have been able to show that the wild-type (WT) -syn gives a bimodal distribution of Rg and Dmax whose relative proportions are varied in the three pathological single point mutations. Residual dipolar couplings (RDCs) determined by 14N1H-HSQC NMR for the WT have been useful in explaining the role of long range interactions in folding, but have not been applied to understanding the behaviour of the familial mutants. To study the familial mutants and those yet to be discovered, amino acid replacement scanning of the whole -syn sequence to determine possible misfolding "hot spots" and perform SAXS-EOM and 14N1H-HSQC NMR would be a huge task. However, it has been shown that it is possible to simulate RDCs from the sequence of intrinsically disordered proteins using the Flexible Meccano and Pales software. In this presentation, we shall show how simulated RDCs, validated by our historic SAXS data can suggest regions where changes in long and short range interactions can lead to misfolding. Thus forearmed, we can tackle the challenges of experimental validation.

Keywords or phrases (comma separated):

SAXS, EOM, Parkinsons Disease, residual dipolar couplings

Welcome Function, Poster Session, Exhibition - Board: 902 / **112**

FTIR detection of different phases of fatty acids forming 3D-assemblies

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Self-assembly of molecules on highly oriented pyrolytic graphite (HOPG) to form ordered patterns have been an area of active research since it is relevant to colloidal stabilization, patterning and thin film devices. Most of the works have focused on 2D crystalline layers of the molecules absorbed onto graphite surfaces. However, more and more research has provided the significance of forming 3D-structures onto surfaces, for instance, superhydrophobicity, self-cleaning and bactericides are induced by 3D wax crystals present on natural surfaces such as plant leaves and insect wings. Two fatty acids, palmitic acid and stearic acid, which are ubiquitous in many organisms, have been found to be the main contributors in the forming of 3D structure of Hemianax papuensis dragonfly wings. Therefore, understanding and mimicking the formation of 3D-patterns in this case is not only important to biological process but also to potential applications. In this work, the two fatty acids were self-assembled onto the surface of HOPG which produced ordered 3D-assemblies. However, despite their similarities in chemical properties, their 3D structure appeared greatly different. Palmitic acid formed into 3D-microblades whilst stearic acids formed into shorter plates. In order to understand the mechanism of such variations, Synchrotron FTIR in ATR mode was employed. Peak shifts in CH2 νas peaks were observed between the two fatty acids. It is postulated that this might be due to different phases present within the same 3D-assemblies of the two fatty acids.

Keywords or phrases (comma separated):

graphite, insect wing, fatty acids, self-assembly

Welcome Function, Poster Session, Exhibition - Board: 107 / **113**

Hydrogen Bonding of O-Ethylxanthate Compounds and Neutron Structural Determination of C–H•••S Interactions

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The hydrogen bonding in mixed sulfur/oxygen acceptor systems can be thoroughly investigated using the O-ethylxanthate (or O-alkyldithiocarbonate) family of anions. A series of O-ethylxanthate salts (guanidinium, methylammonium, dimethylammonium, trimethylammonium, tetramethylammonium, tetraethylammonium, and tetrapropylammonium), were structurally characterised using synchrotron X-ray spectroscopy to demonstrate the influence of the cation on the overall packing of the salts into either 3-D, 2-D or 1-D hydrogen-bonded arrangements. The protic cations vary in the number of available hydrogen bond donors which in turn affects the dimensionality of the hydrogen-bonded networks that form. The guanidinium cation gives rise to a 3-D hydrogen-bonded network due to the large number of NH hydrogen bond donors, whereas the use of ammonium cations, of the nature MexH4–xN+ (x = 1 – 3), demonstrates the decreasing dimensionality of the structures as the number of hydrogen bond donors decreases. Aprotic cations were also studied to demonstrate the influence of C-H…S interactions on the overall packing arrangement of the structure, in the absence of strong hydrogen bond donor groups. Laue neutron diffraction data was used to locate weak C-H…S hydrogen bonds in (Me4N)(EtXn) through the location of the exact positions of the CH hydrogen donors. The neutron data demonstrates the unequivocal presence of CH•••S hydrogen bonding, with the H•••S distance significantly shorter than the sum of the van der Waals' radii (shortest interaction 2.67 Å compared with 3.00 Å).

Keywords or phrases (comma separated):

hydrogen bonding, neutron diffraction, sulfur/oxygen acceptor systems

Welcome Function, Poster Session, Exhibition - Board: 403 / **111**

A combined XAS and TEM study on functional cobalt oxide catalysts for water oxidation catalysis

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One of the biggest challenges of the 21st century is to develop methods of producing cheap, carbon-neutral, clean energy.(1) The Sun is a forefront renewable energy source, however current solar technologies are limited by the Sun's diurnal nature. To become a viable future technology, solar energy systems will need to efficiently convert sunlight into energy, and then provide a method of storing this harnessed energy. Photocatalytic water splitting has been considered an attractive way to store solar energy. The reaction product (molecular hydrogen) is an energy dense molecule that be used directly as a clean fuel, or be readily converted into other energy dense materials, such as solar fuels.(2) Solar fuels can be made economic through the use of inexpensive, Earth-abundant materials in the catalytic water oxidation reaction. Some of the most promising candidates for this purpose include phosphate doped metal oxides.(3) Understanding the role of the phosphate dopant in these catalysts is analytically challenging as the active dopant component is present in low levels and results in disordered amorphous materials. Our work focusses on developing a synthetic approach to systematically alter the phosphate dopant in metal oxide catalysts and developing new analytical approaches to understand how the resulting disordered structure correlates with the high catalytic function.

Keywords or phrases (comma separated):

metal oxide, catalyst, renewable energy

Welcome Function, Poster Session, Exhibition - Board: 108 / **117**

Ion Track Formation in Silicon Oxynitrides by Swift Heavy-Ion Irradiation

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Co-author(s): Allina Nadzri [2] ; Daniel Schauries [3] ; Mark Ridgway [4] ; Patrick Kluth [5]

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Amorphous silicon oxynitrides (SiOxNy) are commonly used as barrier material due to their interesting mechanical and chemical properties. However, their application as gradient-index materials makes them also suitable candidates for the synthesis of nanostructures [1].

Here, we present direct evidence for the formation of ion tracks in 1-micron-thick silicon oxynitrides of different stoichiometry. The samples were irradiated with 185 MeV Au13+ ions to create the ion tracks. At such energies, the incident ion interacts predominantly with the system in the electronic regime. The subsequent transfer of energy to the lattice can yield melting along the ion path. While in crystalline materials the rapid quenching freezes in structural disorder resulting in an ion track, in amorphous materials a more complex process takes place [2]. The stoichiometry was determined using spectral Reflectometry and Rutherford backscattering (RBS), while the morphology was characterised by means of Small Angle X-ray Scattering (SAXS) and Fourier Transform Infrared Spectroscopy (FTIR). SAXS measurements indicate a core-shell structure for the ion tracks, with a typical radius between 3-7 nm, following a trend with N content.

[1] Baak, T., Silicon Oxynitride; a material for GRIN optics, Appl. Opt. 21 6 1069 (1982)

[2] Kluth, P. et al., Fine Structure in Swift Heavy Ion Tracks in Amorphous SiO2, Phys. Rev. Lett. 101 175503 (2008)

Keywords or phrases (comma separated):

swift heavy ion irradiation, ion tracks, silicon oxynitrides

Welcome Function, Poster Session, Exhibition - Board: 1005 / **114**

Structural Studies of Streptolysin O from Streptococcus pyogenes

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Cholesterol-dependent cytolysins (CDCs) constitute a family of bacterial toxins that form pores in many cell types. CDCs are secreted as water-soluble monomers, bind to cholesterol-rich membranes, oligomerise and insert into cell membranes. The presence of membrane cholesterol is required for the formation of large pores in cell membranes. In order to convert from a soluble monomeric protein into a membrane pore conformational changes of the three-dimensional structures of these toxins have to occur. Here, we present the three-dimensional structure of streptolysin O (SLO) from Streptococcus pyogenes. Comparison with other CDCs structures shows that the overall fold is similar but the C-terminal domain exhibits a different orientation with respect to the rest of the molecule. Additionally, the highly conserved region called the undecapeptide motif, which is involved in membrane recognition, adopts a different conformation in SLO compared to perfringolysin O (PFO), although the sequences in this region between the two toxins are identical.

Keywords or phrases (comma separated):

bacterial toxins, cholesterol dependent cytolysin

Welcome Function, Poster Session, Exhibition - Board: 801 / **52**

A Compton spectroscopy technique for quality assurance of synchrotron based stereotactic radiotherapy

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Spectroscopy has the potential to be a powerful tool for the quality assurance (QA) of radiotherapy beams; however, direct measurement using spectroscopy detectors is confounded by pulse pile up effects. This is particularly significant for high dose rate, synchrotron based stereotactic radiotherapy modalities such as microbeam radiation therapy (MRT). We herein investigate a Compton spectroscopy technique to infer the energy spectrum of the primary beam by measuring energies of photons scattered through 90 degrees in air. Compton spectroscopy of an MRT beam was performed using a collimated Amptek CdTe detector at the Imaging and Medical Beamline (IMBL) of the Australian Synchrotron. The response of the system as a function of energy was determined both experimentally, using a monochromator in the energy range 30-90 keV, and by simulation using the Geant4 Monte Carlo toolkit for x-ray energies between 10-300 keV. This response function, along with the Compton equation, can be used to reconstruct the incident energy spectrum for subsequent comparison with the theoretically predicted energy spectrum.

Keywords or phrases (comma separated):

Spectroscopy, Microbeam Radiation Therapy, Radiotherapy, Quality Assurance

Welcome Function, Poster Session, Exhibition - Board: 503 / **50**

Formation of embedded SiGe alloy nanoparticles in Si3N4

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The structural properties of Ge NPs synthesised by ion implantation in amorphous Si3N4 at 400 oC. A combination of conventional techniques (XRD and RBS) and synchrotron-based method have been used to investigate the properties of NPs. XRD spectra reveals poly crystallization of the matrix for samples annealed at 1100 oC and a peak related to SiGe structure. RBS study indicates diffusion of Ge atoms toward the Si substrate after annealing at 1100 oC. In addition, X-ray Absorption Spectroscopy quantified the interatomic structure of implanted samples both as function of concentrations, and post annealing. The formation of a Si(1-x)Gex NPs with different compositions related to different atomic concentration of Ge ions, for all examined samples was readily evident. Regardless of implantation fluence all samples implanted at 1100 oC result in poly crystallization of the

matrix and therefore formation of crystalline SiGe NPs. However, for other annealing temperatures no extended structures formed indicating that the SiGe NPs are in amorphous phase.

Crystallization of the nitride matrix enables the rapid diffusion of Ge atoms to the Si/Si3N4 interface. we discuss the role of implantation and annealing on the growth of NPs in a Si3N4 matrix and compare to that previously observed for Ge in SiO2. We find that implantation and defects appear to contribute to the final structure. The complex mechanisms responsible for crystallisation of the matrix including consideration of structural disorder and non-stoichiometry have been identified.

Keywords or phrases (comma separated):

ion implantation, semiconductor nanoparticles, EXAFS

Welcome Function, Poster Session, Exhibition - Board: 504 / **51**

Temperature dependency analysis of Ge+1 ions embedded in Si3N4 by ion implantation

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A uniform ion distribution of Ge+1 ions were achieved through multiple-energy/fluence implantations of Ge ions into 2μm a-Si3N4, which were grown on Si(100) substrates. Implantations were performed at temperatures of -196, 200 and 400 oC, to investigate the effect of implanting temperature on the phase of the matrix.
Multiple techniques were used to characterise the evolution of the structural properties of samples. The crystalline and amorphous components both as a function of implantation temperature and concentrations, and post-implant annealing were quantified by X-ray Absorption Spectroscopy. The formation of a Si-Ge bonding environments, for all examined concentrations and temperatures was readily evident. For samples implanted at -196 oC, second and third nearest neighbor peaks were observed, indicative of crystalline environment. For samples implanted at higher temperatures, however, there was no extended structure indicating that the Ge environment is amorphous for these samples.
Raman spectroscopy measurements confirmed the implantation-temperature dependent structure. Crystallisation of the nitride matrix enables the rapid diffusion of Ge atoms to the Si/Si3N4 interface. The formation of a thin, non-uniform GexSi(1-x) layer ensued, accompanied by interfacial faceting to relative strain. We find that both implantation and chemical-induced defects appear to be responsible for the various structures that ensue with processing conditions. I have isolated the complex mechanisms responsible for crystallisation of the matrix, including consideration of structure disorders, loss of N2, and non-stoichiometry.

Welcome Function, Poster Session, Exhibition - Board: 601 / **55**

Developments in Partially Coherent Ptychography

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Co-author(s): Bo Chen [1] ; Brian Abbey [1] ; Grant van Riessen [1] ; Keith Nugent [1]

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Ptychography has rapidly developed into a widespread technique for high-resolution X-ray microscopy due to improvements in image quality and the added flexibility over conventional Coherent Diffractive Imaging (CDI) techniques. These benefits are achieved via scanning the sample across a finite incident beam such that overlapping regions reinforce the solution for the sample transmission function in the diffraction data. Recent developments in position correction algorithms ameliorate

the problems associated with sample stage drift and hysterises during image acquisition and reduce drift artefacts. Nevertheless, its application to table-top sources has been less rapid due to their limited coherent flux. Such an extension is vital if diffraction microscopy is to become a widespread and routine imaging modality. In this talk, we present recent results of ptychographic diffractive imaging using partial spatial and temporal coherence and suggest a new condition for the optimal overlap parameter when imaging using partially coherent radiation. This has important implications for users wishing to apply ptychography to conventional sources that may not meet the stringent coherence requirements for conventional diffractive imaging.

Keywords or phrases (comma separated):

ptychography, coherent diffractive imaging, partial coherence, overlap parameter

Welcome Function, Poster Session, Exhibition - Board: 1010 / **128**

Pipeline for in cellulo structure determination

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Co-author(s): Chan Lay [1] ; Fasseli Coulibaly [1] ; Tom Caradoc-Davies [2]

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The production of diffraction-quality crystals remains the major bottleneck in X-ray crystallography, as shown by data from the main structural biology consortia. By contrast, in certain systems, crystals grow readily in the complex environment of the cell used to express the protein, be it in the natural context or in a recombinant system for overexpression. Recent interest in these in vivo crystals have
emerged in the context of a growing impact of microcrystallography brought by serial microcrystallography at synchrotron and X-ray free electron laser facilities.

Our aim is to set up a pipeline to streamline in cellulo diffraction, direct exposure of crystals to X-rays directly through the cells. Using in vivo crystals of the recombinant cypovirus polyhedrin, we show that crystal-containing cells could be selectively sorted by flow cytometry based on their higher side scattering. Crystal-containing cells were dyed with Trypan blue to achieve better visualisation, mounted on micromeshes and flash-frozen. Analysis of these cells on the MX2 beamline of the Australian Synchrotron show that high-quality diffraction data can be collected from in cellulo crystals. The structure of the polyhedrin protein determined by molecular replacement closely matches the model previously determined from purified microcrystals. Advantages of this approach over conventional crystallography and future developments will be discussed in the presentation.

Keywords or phrases (comma separated):

In vivo crystals, in cellulo diffraction

Welcome Function, Poster Session, Exhibition - Board: 109 / **127**

Formation of nanostructures in Silicon Oxynitrides by Ion Implantation

Author(s): Pablo Mota Santiago[1]

Co-author(s): Felipe Kremer [1] ; Mark Ridgway [1] ; Patrick Kluth [1]

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The photoluminescence signal of Amorphous silicon oxynitrides can be tunable by controlling their stoichiometry. The change in PL is related to defect centres and phase structures changes [1]. A coupling between the surface plasmon resonace of Au nanoparticles with these PL centres would lead to develop new optoelectronic and light source devices.

To study this process we implanted 2 MeV Au ions at room temperature with a fluence of 5 X 1016 ions/cm2, following a 60 minute thermal annealing at different atmospheres and temperatures between 1000℃ to 1100 ℃. The different size distributions were determined via Small Angle X-ray Scattering, while the structural parameters were determined by EXAFS [2]. As a result, Au nanoparticles with an average radius between 1-8 nm were found, where the size distribution showed a strong dependence with N concentration.

[1] Huang R. et al., Bright red, orange-yellow and white switching photoluminescence from silicon oxynitrides films with fast decay dynamics, Opt. Mat. Express 4 2 205 (2014)
[2] Giulian R., et al., The influence of annealing conditions on the growth and structure of embedded Pt nanocrystals, J. Appl. Phys. 105 2 044303 (2009)

Keywords or phrases (comma separated):

Au nanoparticles, silicon oxynitrides, ion implantation

Welcome Function, Poster Session, Exhibition - Board: 206 / **126**

Developments in sample characterisation at the SXR endstation

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Many of the more demanding surface science experiments at the soft x-ray beamline are often concerned with the interaction between ordered substrates of novel materials and adlayer molecules or thin films. Whilst soft x-ray spectroscopy is extremely powerful in characterizing the chemical information at such interfaces, being able to measure concomitant changes in other physical properties can provide a more complete story; for example, understanding how we can control free charge carriers in novel materials using molecular acceptor or donor molecules. We detail two major hardware upgrades on the endstation for additional sample characterization. The first has been the addition of an Ultra high Vacuum (UHV) four point probe, capable of measuring the resistivity of samples in-situ. Under a continuous UHV environment, we can now obtain electronic structure and chemical information using soft x-ray spectroscopy, with changes in the electrical properties i.e basic transport measurements. The hardware is currently being extended to measure Hall Bar geometries with back gating. Recent results on DNA nucleobase molecules and their interaction with graphene are shown. The second major initiative has been the to develop robust means of measuring the sample work-function, a property highly sensitive to surface chemistry. The photoemission-based method, using the secondary electron cutoff (SECO), has been initiated. For materials where the SECO method does not work (e.g insulators) we have installed a non-contact UHV Kelvin Probe. Recent results from the Kelvin probe and SECO on functionalised diamond surfaces are presented.

Keywords or phrases (comma separated):

work function, photoemission, Kelvin probe, surface science, graphene

Welcome Function, Poster Session, Exhibition - Board: 604 / **125**

X-ray Fourier-transform holography with customizable references

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Holographic references can enhance the robustness of coherent diffraction imaging experiments and greatly simplify data analysis. However, to date holography has only only been possible with a limited set of special reference waves. We present a new approach to x-ay Fourier-transform holography with an almost unrestricted choice for the reference wave, opening up new avenues to optimize signal-to-noise and resolution. Two geometries that exploit this flexibility are to have the reference and object in separate planes and to have a reference that fills the field of view. Both of these advantages featured in an experiment we performed at the Australian Synchrotron where we explored the potential of our method. In future experiments, our goal is to optimize holography for robustness and resolution by optimizing the reference design to help establish holography as a reliable, accessible, high-resolution coherent imaging technique.

Keywords or phrases (comma separated):

X-ray holography, Coherent diffractive imaging

Welcome Function, Poster Session, Exhibition - Board: 1001 / **123**

Design and Implementation of an Optical Ptychographic Microscope at La Trobe University

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Ptychography is a method for quantitatively determining the phase of of a samples' complex transmission function. The technique relies upon the collection of multiple overlapping coherent diffraction patterns from laterally displaced points on the sample. The overlap of measurement points provides complementary information that significantly aids in the reconstruction of the complex wavefield exiting the sample. Moreover the method is sufficiently robust to simultaneously recover both the sample and probe functions from a single dataset.

Ptychography was initially realised for applications involving electron microscopy (Hoppe et al., Acta Cryst. A, 1969) but has been widely adopted by the x-ray lensless imaging community. More recently, it has found application in the optical regime (e.g. Godden et al. Optics Express, 2014) where it can be applied to 2D and 3D quantitative phase contrast imaging of weakly interacting specimens.

Here we describe and demonstrate the realisation of a high-quality optical ptychographic microscope at La Trobe University comprising 'off the shelf' components. As well as providing important proof-of-principle data for developing coherent imaging experiments at synchrotron and X-ray Free Electron Laser (XFEL) sources, we are planning to develop this instrument for applications involving complimentary live cell imaging.

Keywords or phrases (comma separated):

Optical, Ptychography, CDI, Phase,

Welcome Function, Poster Session, Exhibition - Board: 703 / **121**

Opportunities for Industrial Engagement and Commercial Applications of X-ray and Correlative Microscopy at the Australian Synchrotron

Kathryn Spiers[1]

[1] *Australian Synchrotron*

The Australian Synchrotron is a powerful scientific tool offering state of the art techniques and is ideally positioned to provide services for the evolution and commercialization of the next generation of high-tech products. The increasing pressure to move products to market quickly has required industry to look for new approaches in their development cycle. To this end, industry is embracing the advantages provided by the Synchrotron, with respect to both the technical services and scientific experience and expertise provided to commercial clients.
The world class X ray and Infrared microscopy facilities at the Australian Synchrotron provide unique opportunities to commercial enterprises not available at any other facility in Australia. The large area scanning and high sensitivity of the X ray Fluorescence Microscopy beamline enables the acquisition of high definition elemental maps for industries including agriculture, minerals and mining, and advanced materials. The Infrared Microspectroscopy beamline combines the high brilliance and collimation of the Synchrotron source to achieve a spatial resolution that has been applied to problem solving - such as contaminant identification in manufactured products. The Imaging and Medical beamline ability to produce rapid, time-resolved, high-contrast 3D tomographic images of soft and hard materials has been applied to investigations of pore networks and connectivity in additive manufacturing products and geological systems. Examples will be provided highlighting the strengths of these techniques and the particular benefits they can provide Australian industry.

Keywords or phrases (comma separated):

Industry, Agriculture, Mining, Minerals, Materials

Welcome Function, Poster Session, Exhibition - Board: 202 / **28**

High resolution x-ray beam dosimetry using radiochromic films

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The use of radiochromic film for clinical dosimetry is well established, and in principle these films can provide the high spatial resolution dosimetry required for the microbeam x-ray radiotherapy research taking place on IMBL. The spatial resolution of a measurement made with the radiochromic film is typically limited by the densitometry. For broad beam illuminations (> 1 mm) the spatial resolution of photographic quality commercial scanners has been found adequate. However for the higher resolutions required for microbeam radiation therapy (MRT) where beam dimensions are typically 25 microns, the modulation transfer function (MTF) of such scanners has been proven not to be sufficient.
A densitometry method based on using a microscope with a digital imaging system is potentially both rapid and efficient. We have assessed the use of the IMBL inverted microscope which is equipped with a motorised stage and a digital camera, and devised a potential protocol for high resolution film sensitometry and dosimetry.

Keywords or phrases (comma separated):

Dosimetry, Radiochromic Film, Micro beam radiotherapy

Welcome Function, Poster Session, Exhibition - Board: 402 / **20**

Applications of in situ X-ray powder diffractionto geosciences.

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The powder diffraction beamline at the Australian Synchrotron exploits the unique properties of synchrotron radiation by offering tunable wavelengths (6 keV – 30 keV) to minimise sample absorption, high flux and good S/N for increased detection limits, and high resolution to minimise peak overlap. The X-ray powder diffraction beamline produces bright, high collimated X-ray beams that, when combined with the state-of-the-art Mythen II microstrip detector, are ideal for time-resolved X-ray powder diffraction experiments requiring high resolution data collection. The beamline possesses a vast arsenal of sample stages and environments that enable a multitude of in situ experiments where temperature, pressure or gaseous environment, to name a few, can be varied to observe structure change and/or formation in polycrystalline materials. Since beginning user operations in 2008, over 300 user experiments have been performed at the powder diffraction beamline.

This work demonstrates the capabilities offered by the beamline, particularly for earth sciences and energy applications, highlighting a range of unique in situ experiments that have not only produced successful scientific outcomes, but have pushed the boundaries in many cases. The options for future developments at the powder diffraction beamline are also discussed and will enable the beamline to build on its strong in situ foundations to offer more power and flexibility for its users.

Keywords or phrases (comma separated):

in situ, powder diffraction, synchrotron, beamline

Welcome Function, Poster Session, Exhibition - Board: 407 / **26**

Infrared spectroscopic studies of amorphous ice nanoparticles

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Ice plays an important role in the atmosphere of earth and the interstellar medium through interaction with radiation and hosting chemical reactions. Ice aerosols in the troposphere scatter and absorb radiation from sun and thus have substantial influence on the temperature of earth. Understanding ice's behaviour is believed to be essential for predicting the future of earth.
Due to its suitability for remote sensing Infrared Spectroscopy can be used to probe the physics and chemistry of aerosols in astrophysical and atmospheric context. This has been demonstrated by the data collected using satellite instruments, especially in the Far-IR region. Accurate laboratory measurements are needed in order to interpret these data.

In this study, the spectrum of amorphous ice nanoparticles of submicron size in the 10 – 4000 cm-1 spectral range is investigated utilising the Infrared Spectrometer at the Australian Synchrotron. The optimum condition to generate amorphous ice nanoparticles will be presented followed by discussions of change of spectral features with respect to phase, size and temperature.

Keywords or phrases (comma separated):

IR Spectroscopy

Welcome Function, Poster Session, Exhibition - Board: 404 / **24**

Scanning photoelectron microscopic (SPEM) examination of sulfur evolution on acid leached chalcopyrite with and without added pyrite or soluble iron

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Chalcopyrite (CuFeS2) is the most abundant copper-bearing mineral. The dissolution of chalcopyrite in mine waste environments is considered to contribute to the serious environmental issue of acid and metalliferous drainage (AMD) through release of toxic aqueous copper, particularly in the presence of microbes, dissolved O2, aqueous Fe3+ and pyrite, with the latter two being associated with enhanced chalcopyrite dissolution rates. However, to date few studies have attempted to show the spatial distribution of the surface species formed as well as their evolution as means to better understand the dissolution mechanisms. High resolution, both in terms of energy and spatially, scanning photoelectron microscopy (SPEM), has been applied to the evolution of sulfur species on chalcopyrite surfaces leached in pH 1.0 HClO4 solution at 650 mV (SHE) and 75 ℃ for 5 - 10 days, in the absence and presence of pyrite or with added aqueous iron. Bulk S2-, S22- and Sn2- were found to be present on all samples and oxidation was observed to take place heterogeneously at the sub-micron scale. As compared to chalcopyrite leached for 5 days, surface oxidation did not appear to be increased on extended dissolution to 10 days, however surface roughness increased markedly. Both S0 and SO42-, but no SO32-, were observed when 4 mmol soluble iron was added indicating greater oxidation occurred with greater Fe3+ activity. The greatest surface oxidation was observed when chalcopyrite was in contact with pyrite due to formation of a galvanic couple, with S0, SO32- and SO42- being identified.

Keywords or phrases (comma separated):

SPEM; chalcopyrite; oxidation; dissolution; sulfur species, galvanic interaction

Welcome Function, Poster Session, Exhibition - Board: 207 / **135**

Soft X-ray Imaging by Coherent Diffraction Methods at the Australian Synchrotron

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Co-author(s): Andrew Peele [2] ; Giang Tran [1] ; Mark Junker [1] ; Michael Jones [2]

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[2] *Australian Synchrotron*

Coherent diffractive imaging is a high-resolution method capable of providing phase, chemical and magnetic sensitivity over a large field-of-view[1]. Because iterative algorithms are substituted for

image-forming lenses, the technique is not limited by the difficulty of manufacturing X-ray optics. It has been widely adopted by the international synchrotron community and is quickly becoming a routine imaging and tomography technique across a wide field of applications.

A flexible soft X-ray imaging facility was implemented at the Australian Synchrotron early in 2013 on a dedicated branch of the Soft X-ray Spectroscopy beamline[2]. In the first full year of operation, the Soft X-ray Imaging (SXRI) branchline has been used for a wide range of studies, including imaging fully hydrated, unstained mammalian cells, in situ spectrochemical imaging during electrochemical deposition, and for imaging the domain structure of magnetic thin-films. It has also been used to develop methods of ptychography and holography using partially spatially coherent and partially temporally coherent (polychromatic) illumination.

This talk will first briefly describe the imaging techniques supported at SXRI and the history of their development. Examples of research at SXRI will then be described to illustrate the wide potential for the development and application of these techniques.

1. NW Phillips, ,CT Putkunz, G van Riessen, HD Coughlan, MWM Jones, B Abbey, International Journal of Materials Research, 105, 655-663 (2014)

2. G van Riessen, M Junker, NW Phillips, AG Peele. Proc. SPIE 8851, 885117 (2013).

Keywords or phrases (comma separated):

coherent diffractive imaging, imaging, ptychography, coherence

Welcome Function, Poster Session, Exhibition - Board: 208 / **136**

New laser applications on the THz/FarIR beamline at the Australian Synchrotron

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[2] *The Australian Synchrotron*

Currently, lasers are being introduced to the THz/FarIR beamline at the Australian Synchrotron. This will allow some new techniques such as steady state pump probe, photolysis and pyrolysis experiments to be undertaken at the beamline. We currently have a high powered cw CO2 laser and a pulsed YAG laser.
At the THz beamline, an Enclosive Flow Cooling (EFC) cell is available for use. The EFC cell is a White type cell with a nominal optical path length of 625mm; it can be cooled either with liquid helium or liquid nitrogen, or can be operated up to 400K with a temperature stability of ±1K per day. It is usually operated under vacuum (~10-3 mbar) but can be pressurized up to 2000 mbar. Cooling not only simplifies complex molecular spectra but also enables the generation of molecular clusters. The capabilities of the cooling cell will be further developed by adding multiple laser sources, thereby allowing a host of sunlight driven reactions to be studied and providing a source of radicals such as OH or halogens.
For species with half lives of the order of fractions of a second or more, thermal or photolytic breakdown of a gas stream containing specifically designed recursor molecules has been successful in previous experiments.
Sunlight driven reactions are incredibly important for atmospheric studies. An example of this is so that we can understand processes relevant to ozone formation and depletion and investigate various aerosols that contribute to this.

Keywords or phrases (comma separated):

THz, FarIR, laser, photolyisis, pyrolysis

Welcome Function, Poster Session, Exhibition - Board: 602 / **131**

Correlative hydrated cellular imaging using Coherent Diffraction Imaging at the Australian Synchrotron

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[5] *Australian Synchrotron*

X-ray coherent diffractive imaging provides high resolution, high sensitivity images of intact cellular specimens without the need for sectioning, staining, or tagging. Recent advances in this field allow high resolution imaged to be obtained with a fraction of the dose than otherwise possible while increasing image quality. Further advances have pushed the technique into the X-ray water-window, opening up the possibility to image intact cellular specimens in their native hydrated environment.

Here we present recent results of hydrated cellular coherent diffractive imaging, showing an unprecedented level of image quality and detail. We also outline details of the sample preparation and mounting methods for correlative imaging, together with opportunities for future development.

Keywords or phrases (comma separated):

Ptychography, Cellular Imaging, Correlative Imaging

Welcome Function, Poster Session, Exhibition - Board: 607 / **132**

Experimental Recovery of Sample And Coherence Information in Coherent Diffractive Imaging

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[2] *Australian Synchrotron*

Coherent diffractive imaging (CDI) is a powerful method for recovering the transmission function of an object from its far-field diffraction pattern using iterative algorithms [1]. Recently, it has been shown that CDI works with partially coherent beam [2]. Methods have been developed for dealing with CDI data for which the coherence properties of the illumination are unkown [3]. In this work, we develop a method to simultaneously recover the object's phase and characterise the coherence properties of the illuminating wavefield without any a priori knowledge. The validity of our method is demonstrated using experimental diffraction data from the Soft X-ray Imaging beamline.

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[2] L. W. Whitehead, G. J. Williams, H. M. Quiney, D. J. Vine, R. A. Dilanian, S. Flewett, K. A. Nugent, A. G. Peele, E. Balaur, and I. McNulty, "Diffractive Imaging Using Partially Coherent X Rays", Phys. Rev. Lett. 103, 243902 (2009).
[3] Pierre Thibault, Andreas Menzel, " Reconstructing state mixtures from diffraction measurements", Nature, Vol 494, 68-71, 07 February 2013.

Keywords or phrases (comma separated):

coherent diffractive imaging, Fresnel coherent diffractive imaging, partial coherence

Working with Industry / **141**

Discussion

Working with Industry / **140**

Hospira at the synchrotron: A partnership in problem solving

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[1] *Hospira*

Hospira is a global leader in the manufacture of generic injectable pharmaceuticals. A case study will be presented showcasing the role of the Australian Synchrotron in a recent Hospira investigation.

Working with Industry / **122**

Connecting Academia with Industry: Some success stories

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The Australian Synchrotron is continually growing its Industrial customer base. The Synchrotron's Industry Group has been providing expert support and facilitation between these Industrial clients and collaborators from more traditional academic and research institutes. These collaborations exist across a diverse and expanding range of fields. This presentation will showcase some of the successful Industrial collaborations incorporating the Synchrotron. Of these, one example is the examination of elemental segregation in heavy rail by X-ray fluorescence microscopy, conducted with the Institute of Rail Technology at Monash University as part of their collaborations with their industrial partners. This study revealed segregation information to a resolution not previously available to the heavy rail industry, and has great potential for future assessments of rail quality and longevity.

Working with Industry / **137**

Applications of Synchrotron Light in Inkjet Technology

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Memjet is a privately held technology company that develops printheads and associated technology for high-speed, low cost digital colour printing. The Memjet Waterfall Printhead Technology comprises a page width printhead made up of 70,400 nozzles, which can continuously fire up to 700 million drops per second. A typical A4 page can be printed in one pass, without scanning back and forth, in 1.6 seconds at 1600 x 800 dpi.

The nozzle size and density on a Memjet printhead creates special requirements for contamination control and maintenance design. Each nozzle is 31.7 um across and contamination in such small nozzles or, indeed, the ink fluidic channels, will show up as fine streaks on paper when ink flow to nozzles is blocked. This can become a more serious problem if a number of closely spaced nozzles are starved of ink.

Due to the size and location of contaminants in nozzles and fluidic channels it can be a challenge to identify them by routine FTIR. This was true for one particular field problem where identification of contaminants blocking nozzles was required. However, the high brilliance and spatial resolution of synchrotron IR light enabled identification of printhead contaminants. This led to a rapid customer response and a rethink in maintenance operation.

Moreover, FTIR analysis of the silicon oxide roof layer of printheads showed differences in the nature of the layer across the printhead. Indeed, some of these differences correlated with print quality degradation. Follow up work on the WAXS beamline to comprehend the layer structure in more detail found a predictor peak that changed significantly in intensity between good and bad print quality regions. The position of the peak at 2.57 A-1 suggested it came from a crystallographic form of silicon oxide - possibly alpha quartz.

The FTIR and WAXS investigation clearly showed there were differences in surface chemistries across printheads. This information and other supporting data helped improve our understanding of the wetting behaviour across printheads and the impact on print quality.